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# PHASE II SAMPLING AND ANALYSIS PLAN FOR OPERABLE UNIT 3 LIBBY ASBESTOS SUPERFUND SITE

Part B: Ambient Air and Groundwater

Prepared by
U.S. Environmental Protection Agency
Region 8
Denver, CO



With Technical Assistance from:

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and

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#### APPROVAL PAGE

Part B of the Phase II Sampling and Analysis Plan for Operable Unit 3 of the Libby Asbestos Superfund Site is approved for implementation.

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# TABLE OF CONTENTS

1.0	PROJE	ECT OVERVIEW	. 1
1.1 1.2		pose of this Documentect Management and Organization	
2.0	BACK	GROUND AND PROBLEM DEFINITION	. 5
2.1 2.2 2.3	Bas	Description	. 5
3.0	SUMN	MARY OF EXISTING DATA	. 7
3.1 3.2		bient Airundwater	
4.0	DATA	QUALITY OBJECTIVES	. 9
4.1 4.2	Data	erview of the DQO Processa Quality Objectives for Ambient Air	. 9
4.	.2.1 2.2 2.3	State the Problem	10
4.	2.4 2.5	Define the Bounds of the Study  Define the Decision Rule	10 10
	2.6 2.7	Define the Acceptable Limits on Decision Errors  Optimize the Design	
4.3 4.	Data 3.1	A Quality Objectives for Groundwater	12
4.	3.2 3.3	Identify the Decisions	13
4.	3.4	Define the Decision Rule	14
	3.5 3.6	Define the Acceptable Limits on Decision Errors  Optimize the Design	
5.0	SAMP	LING PROGRAM	15
5.1	Amb	oient Air	15
_		Experimental Design	
	1.2	Sample Collection Protocol	
	1.3 1.4	Sample Collection Schedule  Field QC Samples	
	1. <del>4</del> 1.5	Field Documentation	
5.2		undwater	
	2.1	Experimental Design.	
	2.2	Sampling Methods and Procedures	
	2.3	Sample Containers and Preservation	
	2.4	Field OC Samples for Groundwater	

5	.2.5 Field Data Measurements	18
5	.2.6 Field Documentation	18
5.3	Sample Documentation and Identification	18
5.4	Sample Chain-of-Custody and Shipment	19
6.0	LABORATORY ANALYSIS REQUIREMENTS	21
6.1	Analysis of LA in Air or Water	21
	.1.1 Laboratory Qualifications	
	.1.2 Analytical Method	
	.1.3 Stopping Rules	
	.1.4 Holding Times	
6.2	Analytical Methods for Other (Non-Asbestos) Analytes in Groundwater	
	.2.1 Analyte List for Groundwater	
	.2.2 Target Detection Limits	
	.2.3 Holding Times	
6.3	Instrument Calibration and Frequency	
6.4	Laboratory Custody Procedures and Documentation	
6.5	Laboratory Health and Safety	
6.6	Documentation and Records	
6.7	Sample Archival and Final Disposition	
6.8	Data Deliverables	23
7.0	QUALITY CONTROL	
7.1	Field-Based Quality Control Samples	27
7	1.1 Field QC Samples for Air	27
7	1.2 Field QC Samples for Groundwater	
7.2	Laboratory-Based Quality Control Samples	29
7	2.1 Laboratory-Based QC for LA	
7	.2.2 Laboratory-Based Quality Control Samples for Non-Asbestos Analyses	30
7.3		32
7.4	Quality Assurance Objectives For Measurement Data	
7	.4.1 Precision	33
7	.4.2 Accuracy	
7	.4.3 Representativeness	34
7	.4.4 Completeness	
7	.4.5 Comparability	35
8.0	DATA MANAGEMENT	37
8.1	Data Applications	37
8.2	Roles and Responsibilities for Data Flow	37
	.2.1 Field Personnel	
	.2.2 Laboratory Personnel	
	.2.3 Database Administrators	
8.3	Data Storage	
9.0	ASSESSMENT AND OVERSIGHT	
	A concements	30

9.7	I.I	Field Oversight	39
		Laboratory Oversight	
		ponse Actions	
		orts to Management	
		VALIDATION AND USABILITY	
		a Validation and Verification Requirementsonciliation with Data Quality Objectives	
		RENCES	

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# LIST OF TABLES

Table 3-1	Ambient Air Field Sample Results
Table 5-1	Summary of Well Information Provided by MWH
Table 5-2	Sample Container, Preservation and Handling Requirements, and Holding Times
	for Groundwater Samples
Table 6-1	Analyses of Groundwater for Non-Asbestos Parameters
Table 7-1	Summary of Field Quality Control Samples
Table 7-2	Summary of Laboratory Quality Control Measures by Analysis

# LIST OF FIGURES

Figure 2-1	Operable Unit 3
Figure 3-1	Wind Rose for Zonolite Mountain
Figure 5-1	Ambient Air Sampling Locations
Figure 5-2	Well Locations

# LIST OF ATTACHMENTS

Attachment A	Standard Operating Procedures
Attachment B	Laboratory Modification Forms
Attachment C	GPS Coordinates of Ambient Air Sampling Stations

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#### LIST OF ACRONYMS

CAR Corrective Action Request

CCV Continuing Calibration Verification

DQO Data Quality Objective EDD Electronic Data Deliverable

EDXA Energy Dispersive X-Ray Analysis
EPA U.S. Environmental Protection Agency
EPH Extractable Petroleum Hydrocarbons

FS Feasibility Study

FSDS Field Sample Data Sheets
FSP Field Sampling Plan
FTP File Transfer Protocol

GC/MS Gas chromatography/mass spectroscopy

GO Grid opening

HASP Health and Safety Plan

ICV Initial Calibration Verification

ID Identification IL Inter-laboratory

ISO International Organization for Standardization

IS Internal Standard LA Libby Amphibole

LCS Laboratory Control Sample

LCSD Laboratory Control Sample Duplicate

MDEQ Montana Department of Environmental Quality

MS Matrix Spike

MSD Matrix Spike Duplicate

NVLAP National Voluntary Laboratory Accreditation Program

OU Operable Unit

PDF Portable Document Format PE Performance Evaluation

PR Percent Recovery
QA Quality Assurance

QAPP Quality Assurance Project Plan
QATS Quality Assurance Technical Support

QC Quality Control RD Recount Different RF Response Factors

RPD Relative Percent Difference RPM Remedial Project Manger

RS Recount Same

RSD Relative Standard Deviation

SAED Selective Area Electron Diffraction

# LIST OF ACRONYMS (cont.)

SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
TAL	Target Analyte List
TEM	Transmission Electron Microscopy
TWF	Time-Weighting Factor
VOC	Volatile Organic Compound
VPH	Volatile Petroleum Hydrocarbons

# PHASE II SAMPLING AND ANALYSIS PLAN FOR OPERABLE UNIT 3 LIBBY ASBESTOS SUPERFUND SITE

#### PART B: AMBIENT AIR AND GROUNDWATER

#### 1.0 PROJECT OVERVIEW

#### 1.1 Purpose of this Document

This document is Part B of the Phase II Sampling and Analysis Plan (SAP) for Operable Unit 3 (OU3) of the Libby Asbestos Superfund Site. OU3 includes the property in and around the former vermiculite mine and the geographic area surrounding the former vermiculite mine that has been impacted by releases and subsequent migration of hazardous substances and/or pollutants or contaminants from the mine, including ponds, Rainy Creek, Carney Creek, Fleetwood Creek, and the Kootenai River. Rainy Creek Road is also included in OU3. The exact geographic area of OU3 has not yet been defined but will be based primarily upon the extent of contamination associated with releases from the former vermiculite mine as determined in the remedial investigation (RI) of OU3.

The purpose of Part B of the Phase II SAP for OU3 is to guide the collection of data on mining-related contaminants in ambient air and groundwater to assess the impact of releases from the mined area. The complete scope of Phase II is expected to include collection of data on other environmental media of potential concern in OU3. Requirements will be described in subsequent parts of the Phase II SAP for OU3. These data will be used to support an RI of OU3, the goal of which is to characterize the nature and extent of mining-related contamination in OU3, and to characterize the nature and level of risk posed by mining-related contamination to human and ecological receptors in OU3.

This SAP contains the elements required for both a field sampling plan (FSP) and quality assurance project plan (QAPP). This SAP has been developed in accordance with the U.S. Environmental protection Agency (EPA) Requirements for Quality Assurance Project Plans (EPA 2001) and the Guidance on Systematic Planning Using the Data Quality Objectives Process – EPA QA/G4 (EPA 2006). The SAP is organized as follows:

Section 1 – Project Overview

Section 2 – Background and Problem Definition

Section 3 – Summary of Existing Site Data

Section 4 – Data Quality Objectives

Section 5 – Sampling Program

Section 6 – Laboratory Analysis Requirements

Section 7 – Quality Control

Section 8 – Data Management

Section 9 - Assessment and Oversight

Section 10 - Data Validation and Usability

Section 11 – References

#### 1.2 Project Management and Organization

#### Project Management

EPA is the lead regulatory agency for Superfund activities within OU3. The EPA Remedial Project Manager (RPM) for OU3 is Bonita Lavelle, EPA Region 8. Ms. Lavelle is a principal data user and decision-maker for Superfund activities within OU3.

The Montana Department of Environmental Quality (MDEQ) is the support regulatory agency for Superfund activities within OU3. The MDEQ Project Manager for OU3 is Catherine LeCours. EPA will consult with MDEQ as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and applicable guidance in conducting Superfund activities within OU3.

EPA has entered into an Administrative Order on Consent (AOC) with Respondents W.R. Grace & Co.-Conn. and Kootenai Development Corporation (KDC). Under the terms of the AOC, W.R. Grace & Co.-Conn. and KDC will implement this SAP. The designated Project Coordinator for Respondents W.R. Grace & Co.-Conn. and KDC is Robert Medler of Remedium Group, Inc.

#### **Technical Support**

EPA will be supported in this project by a number of contractors, including:

- Syracuse Research Corporation (SRC) will assist in the development of sampling and analysis plans, and in the evaluation and interpretation of the data.
- NewFields Boulder LLC, a contractor to SRC, will provide support in sampling and analysis, mapping and other GIS applications, and design and evaluation of the feasibility study.
- Department of Transportation, John A. Volpe National Transportation Systems Center (Volpe) will provide management and coordination of resources for field oversight of sampling activities.
- Camp Dresser & McKee, Inc. (CDM), a contractor to Volpe, will provide on-site support and oversight for field sampling activities.

#### Field Sampling Activities

All field sampling activities described in this SAP will be performed by W.R. Grace & Co.-Conn. and KDC, in strict accord with the sampling plans developed by EPA. W.R. Grace & Co.-Conn. and KDC will be supported in this field work by MWH Americas, Inc. (MWH) and by their subcontractors. Individuals responsible for implementation of field sampling activities are listed below:

- MWH Project Director: Michael DeDen
- MWH Project Manager: John D. Garr
- MWH Field Quality Control Officer: Mark Rettmann
- MWH Quality Assurance Officer: Stephanie A. Boehnke

#### On-Site Field Coordinator

Access to the mine is currently restricted and is controlled by EPA. The on-site point of contact for access to the mine is Courtney Zamora of the U.S. Department of Transportation, John A. Volpe National Transportation Systems Center (Volpe).

#### Sample Preparation and Analysis

All samples collected as part of the Phase II investigation will be sent for preparation and/or analysis at laboratories selected and approved by EPA.

- All analyses of samples for asbestos will be performed by EMSL Analytical, Inc., or other laboratories approved by EPA.
- All analyses of samples for non-asbestos analytes will be performed by Energy Laboratories, Inc. (ELI).
- All validation and verification activities for asbestos and non-asbestos data will be performed by SRC or their subcontractors.

#### Data Management

Administration of the master database for OU3 will be performed by EPA contractors (SRC and NewFields). The primary database administrator will be Lynn Woodbury. She will be responsible for sample tracking, uploading new data, performing error checks to identify inconsistent or missing data, and ensuring that all questionable data are checked and corrected as needed. When the OU3 database has been populated, checked and validated, relevant asbestos data will be transferred into the Libby2 database or other Libby Asbestos Site database as directed by EPA for final storage.

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#### 2.0 BACKGROUND AND PROBLEM DEFINITION

#### 2.1 Site Description

Libby is a community in northwestern Montana that is located near a large open-pit vermiculite mine. Vermiculite from the mine at Libby is known to be contaminated with amphibole asbestos that includes several different mineralogical classifications, including richterite, winchite, actinolite and tremolite. For the purposes of EPA investigations at the Libby Superfund Site, this mixture is referred to as Libby Amphibole (LA).

Historic mining, milling, and processing of vermiculite at the site are known to have caused releases of vermiculite and LA to the environment. Inhalation of LA associated with the vermiculite is known to have caused a range of adverse health effects in exposed humans, including workers at the mine and processing facilities (Amandus and Wheeler 1987, McDonald et al. 1986, McDonald et al. 2004, Sullivan 2007, Rohs et al. 2007), as well as residents of Libby (Peipins et al. 2003). Based on these adverse effects, EPA listed the Libby Asbestos Site on the National Priorities List in October 2002.

Starting in 2000, EPA began taking a range of cleanup actions at the site to eliminate sources of LA exposure to area residents and workers using CERCLA (or Superfund) authority. Given the size and complexity of the Libby Asbestos Site, EPA designated a number of Operable Units (OUs). This document focuses on investigations at Operable Unit 3 (OU3). Figure 2-1 shows the location of the mine and a preliminary study area boundary for OU3. EPA established the preliminary study area boundary for the purpose of planning and developing the scope of the remedial investigation/feasibility study (RI/FS) for OU3. This study area boundary may be revised as data are obtained during the RI for OU3 on the nature and extent of environmental contamination associated with releases that may have occurred from the mine site. The final boundary of OU3 will be defined by the final EPA-approved RI/FS.

#### 2.2 Basis for Concern

EPA is concerned with environmental contamination in OU3 because the area is used by humans for logging and a variety of recreational activities, and also because the area is habitat for a wide range of ecological receptors (both aquatic and terrestrial). Contaminants of potential concern to EPA in OU3 include not only LA, but any other mining-related contaminants that may have been released to the environment.

#### 2.3 Scope and Strategy of the RI at OU3

As noted above, Respondents W.R. Grace & Co.- Conn. and KDC are performing an RI in OU3 under EPA oversight in order to characterize the nature and extent of environmental

contamination and to collect data to allow EPA to evaluate risks to humans and ecological receptors from mining-related contaminants in the environment.

The RI is being performed in several phases. Phase I of the RI was performed in the fall of 2007 in accord with the *Phase I Sampling and Analysis Plan for Operable Unit 3* (USEPA 2007). The primary goal of the Phase I investigation was to obtain preliminary data on the levels and spatial distribution of asbestos and also other non-asbestos contaminants that might have been released to the environment in the past as a consequence of the mining and milling activities at the site.

Phase II of the OU3 RI is being performed in the spring, summer, and fall of 2008. Phase II is composed of three parts, as follows:

- Part A focuses on the collection of data on the levels of LA and other chemicals of concern in surface water and sediment, as well as site-specific toxicity testing of surface water using rainbow trout.
- Part B focuses on the collection of data on LA levels in ambient air samples collected near the mined area, and on the collection of data on LA and other chemicals of potential concern in groundwater.
- Part C focuses on the collection of other data needed to support the ecological risk assessment at the site.

This SAP is Part B of the Phase II RI. Part A and Part C are described in separate SAPs (USEPA 2008a, 2008b).

#### 3.0 SUMMARY OF EXISTING DATA

#### 3.1 Ambient Air

Based on meteorological data collected at the mine site, the predominant direction of wind flow at the mine is to the northeast (Figure 3-1), so it is expected that current releases and historic impacts are likely to be highest in this direction. However, because of the variability in wind direction, releases and impacts are possible in other directions as well.

As part of the Phase I investigation, a set of eight ambient air monitoring stations were established in the vicinity of the mined area. Four samples were collected from each station, with each sample representing a 5-day composite collected between October 2 and October 22, 2007. The locations of these samples can be found in the Phase I Sampling and Analysis Plan for OU3 (USEPA 2007). The purpose of these samples was to obtain preliminary data on the level of LA that is currently being released from the mine site.

All air samples were analyzed for asbestos by TEM in accord with the ISO 10312 method (ISO 1995) counting protocols, with all applicable Libby site-specific laboratory modifications, including the most recent versions of modifications as specified in the SAP (USEPA 2007). The results of analyses of asbestos in the ambient air samples are provided as Table 3-1. Asbestos was not detected in any of the field samples. These results should be interpreted cautiously because ambient air samples were collected over a time interval when rain was occurring frequently, which may have reduced the potential for airborne releases to ambient air.

#### 3.2 Groundwater

No data were located on measured concentrations of LA in groundwater samples from the site.

Two groundwater samples with limited data on non-asbestos analytes were discovered in the archived results. The first was collected by Shafer and Associates in November 1991 from a station identified as PW-2 that was described as "groundwater near SW-11". The sample was analyzed for field parameters (pH, EC, temp), major ions (sodium, potassium, calcium, magnesium, chloride, bicarbonate, carbonate, fluoride, nitrate, and sulfate), and some metals (arsenic, cadmium, chromium, copper, iron, lead, mercury, nickel, and zinc). The second groundwater sample was collected in March 1986 by W.R. Grace, and was identified as "New Well". This sample was analyzed for field parameters (pH, EC), major ions (sodium, calcium, magnesium, bicarbonate, carbonate, fluoride, nitrate, and sulfate), and some metals (arsenic, barium, cadmium, chromium, iron, lead, manganese, mercury, selenium, and silver).

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#### 4.0 DATA QUALITY OBJECTIVES

#### 4.1 Overview of the DQO Process

Data Quality Objectives (DQOs) define the type, quality, quantity, purpose, and intended uses of data to be collected (EPA 2006). The design of a study is closely tied to its DQOs, which serve as the basis for important decisions regarding key design features such as the number and location of samples to be collected and the chemical analyses to be performed. In brief, the DQO process typically follows a seven-step procedure, as follows:

- 1. State the problem that the study is designed to address
- 2. Identify the decisions to be made with the data obtained
- 3. Identify the types of data inputs needed to make the decision
- 4. Define the bounds (in space and time) of the study
- 5. Define the decision rule which will be used to make decisions
- 6. Define the acceptable limits on decision errors
- 7. Optimize the design using information identified in Steps 1-6

Following these seven steps helps ensure that the project plan is carefully thought out and that the data collected will provide sufficient information to support the key decisions which must be made.

#### 4.2 Data Quality Objectives for Ambient Air

#### 4.2.1 State the Problem

LA fibers may be released into air from the mined area as a result of wind or human disturbances of mine waste or contaminated soils. LA fibers that are released into air are of concern for two reasons: 1) humans who are present in OU3 may be exposed by inhaling the fibers, and 2) fibers in air may be transported by wind and subsequently deposited to soil or plant material, resulting in contamination of media away from the mine.

As noted above, the Phase I investigation sought to collect data on LA levels in ambient air. These samples did not detect LA in any sample, but these data are not considered to be sufficient to conclude ambient air is not of concern for the following reasons:

- The samples were collected during a time of frequent rain, so the potential for release may have been reduced.
- The observations span a time period of only 20 days, while evaluation of human health risk associated with inhalation of LA in ambient air requires information on long-term average concentrations.

Because concentrations of LA in ambient air may vary over time (daily and seasonally), especially in cases where there are fluctuations atmospheric conditions (wind direction and speed, relative humidity, precipitation, etc.), additional data are needed to characterize the levels of LA in ambient air as a function of time as well as space.

#### 4.2.2 Identify the Decisions

The decision that EPA must make is what response actions, if any, are needed to protect human receptors from unacceptable risks from asbestos in ambient air.

#### 4.2.3 Identify the Types of Data Needed

The principal type of data that is needed to evaluate risks from LA in ambient air is reliable and representative measurements of the long-term average concentration of LA as a function of both time and space. In addition, data on wind speed and direction are needed in order to help evaluate the air data. These data are being collected by a meteorological station that has been installed at the site.

#### 4.2.4 Define the Bounds of the Study

#### **Spatial Bounds**

Conceptually, the area of concern for LA in ambient air might be quite large, since LA fibers in air can be transported by wind for long distances. However, the focus of Phase IIB is to monitor the levels of LA in air relatively close to the mine site, since this is where the concentrations are likely to be highest. If levels of LA in air near the mine site are above a level of potential concern, then the investigation of ambient air may be expanded to more fully characterize the locations and materials in the mined area that are the primary sources of off-site release, and the airborne dispersion of the LA particles as a function of distance and direction from the mine.

#### Temporal Bounds

Because the level of release of LA from the mined area into air is expected to vary over time, measurements are needed at multiple time points in order to fully characterize the variability and average concentration values. Releases are not likely to be of concern during winter when the ground is snow-covered, but may be of concern during the spring, summer, and fall when the ground is un-covered and subjected to wind and human disturbance activities.

#### 4.2.5 Define the Decision Rule

EPA has not formally specified the decision rule that will be used to determine if it is necessary to evaluate response actions to address releases of LA into ambient air. However, it is

anticipated that decisions will be based mainly on the estimated level of human health risk from ongoing airborne releases of LA. If the level of release from the site is below a level of health concern, then it is not expected that evaluation of response actions to reduce or eliminate ongoing airborne emissions will be required. If releases from the mine site to off-site air approach or exceed a level of concern to humans, then evaluation of response actions may be required.

#### 4.2.6 Define the Acceptable Limits on Decision Errors

In making a decision regarding the health risks to humans from inhalation of LA in ambient air, two types of error are possible:

- A false negative decision occurs when it is decided the risks are acceptable when the true risks are above a level of concern.
- A false positive decision error occurs when it is decided the risks are not acceptable when the true risks are acceptable.

EPA is mainly concerned with limiting the likelihood of a false negative decision error. For this reason, EPA generally seeks to ensure that there is no more than a 5% chance or a false negative decision error.

EPA is also concerned with the probability of false positive decision errors. Such an error does not result in unacceptable human health risk, but may result in the un-needed expenditure of resources that could be invested more effectively elsewhere. For OU3, EPA will seek to ensure that there is less than a 20-30% chance of a false positive decision error in cases where the true risk is less than ½ the level of concern.

#### 4.2.7 Optimize the Design

#### Estimating the Number of Samples

Human health risk from ambient air is evaluated using the long-term average concentration value. However, there is uncertainty in any such measured value. This uncertainty arises from two sources:

- Variability in the actual air concentrations over time and space. This is usually assumed to be characterized by a lognormal distribution.
- Uncertainty in the accuracy of the measured concentration values of the samples collected. This is characterized by a Poisson distribution.

Because of these two sources of variation, a data set of ambient air samples is expected to be characterized by a Poisson-lognormal (PLN) distribution. The magnitude of the uncertainty around the mean of a PLN data set depends on three key variables:

- as the variability in the underlying lognormal distribution (GSD) increases, uncertainty increases
- as the number of samples collected increases, uncertainty decreases
- as the number of particles counted per sample (lamda) increases, Poisson uncertainty decreases

At present, data are not available to estimate how close the mean concentration of LA in ambient air in OU3 is to a level of concern, or on the magnitude of the underlying variability. In the absence of such data, the minimum number of samples to be collected in this effort is 40. It is expected that this should be sufficient to support decision making if between-sample variability is not too high and if the observed mean concentration is not too close to a decision threshold. Additional sampling may be needed to support decision-making if variability is high and/or observed means are close to decision thresholds.

#### Estimating the Required Analytical Sensitivity

The level of analytical sensitivity needed for ambient air samples depends on the level of human exposure that is anticipated in the vicinity of the mine site. For planning purposes, it is conservatively assumed that the maximally-exposed individual would be a present near the site about 8 hours per day for about 150 days per year, for a total of 25 years out of a lifetime. This corresponds to a time-weighting factor (TWF) of  $(8/24) \cdot (150/365) \cdot (25/70) = 0.049$ . Based on an assumed target risk of 1E-05 ( $1/10^{th}$  the level that is normally considered to be the maximum acceptable total risk to a receptor), and using the unit risk value of 0.23 (f/cc)<sup>-1</sup> currently recommended by EPA (IRIS 2008), the target analytical sensitivity is calculated as follows:

Target Sensitivity = Target Risk / (TWF · Unit Risk)  
= 
$$1E-05 / (0.049 \cdot 0.23)$$
  
 $\approx 0.001 \text{ f/cc}$ 

## 4.3 Data Quality Objectives for Groundwater

#### 4.3.1 State the Problem

Groundwater near the mine site may contain LA and/or other contaminants released as a consequence of past mining and milling activities at the site. Under current land use conditions, it is not believed that humans are exposed to groundwater, but it is considered plausible that groundwater wells might be installed sometime in the future. Likewise, ecological receptors are not generally exposed directly to groundwater, but groundwater may recharge surface water bodies such as streams or ponds, potentially leading to the exposure of aquatic receptors.

At present, data are insufficient to characterize groundwater at the site, either with regard to concentration levels of other mining-related chemicals, or with regard to rate and direction of flow and the location of groundwater discharges to surface.

#### 4.3.2 Identify the Decisions

The decision that EPA must make is what response actions, if any, are needed to protect human and ecological receptors from unacceptable risks from exposure to asbestos and any other mining-related contaminants in groundwater.

#### 4.3.3 Identify the Types of Data Needed

#### Contaminant Concentration Data

One type of data that is needed to evaluate risks from contaminants in groundwater is reliable and representative measurements of the concentration of contaminants as a function of both time and space. This includes not only the concentration of LA, but other chemicals that may have been released into groundwater as a consequence of historic mining and milling activities at the site. The list of analytes required for groundwater is presented in Section 6.2.

#### Groundwater Elevation and Flow

A second type of data needed to evaluate groundwater at the site is measurements of groundwater elevation and gradient characteristics. This is needed to evaluate the extent to which any contaminants in groundwater may be migrating away from the mined area, and the potential that contaminated groundwater may recharge surface water bodies where exposure of humans or ecological receptors may occur.

#### **Spatial Bounds**

For the purposes of the Phase IIB investigation, groundwater will be investigated through the use of existing wells that are located within OU3, on or near the mined area. The location of these wells is presented in Section 5.2.

#### Temporal Bounds

Variation in the concentration of contaminants in groundwater may occur as a function of time season as groundwater levels and flows vary between seasons. Therefore, for initial characterization of groundwater conditions, three separate groundwater monitoring events are required to characterize variations in groundwater elevations, groundwater flow directions, and groundwater quality associated with the seasonal variability in local precipitation, groundwater recharge, and surface-water flow conditions. Groundwater monitoring will take place during

mid-summer and fall of 2008 and early spring of 2009. The mid-summer monitoring event will coincide with surface water sampling conducted as part of Element 1 of the Phase IIA remedial investigation described in the Phase IIA Sampling and Analysis Plan for OU3 dated March 20, 2008 (USEPA 2008a). The projected date for this event is the week of August 18, 2008. The late summer monitoring event will occur during the week of September 29, 2008. The late spring monitoring event will occur in May, 2009.

#### 4.3.4 Define the Decision Rule

EPA has not formally specified the decision rule that will be used to evaluate groundwater at OU3. However, it is anticipated that if health risk from hypothetical future human ingestion of groundwater is unacceptable for one or more exposed populations, or if groundwater impacts to surface water are judged to be of potential concern to humans or ecological receptors, then it will be concluded that it is appropriate to evaluate response actions to reduce contamination, exposure, and/or migration of groundwater at the site.

#### 4.3.5 Define the Acceptable Limits on Decision Errors

As discussed above, at OU3 EPA will seek to limit the chances of a false negative decision error to 5%, and to limit the probability of a false positive decision error to about 20-30% when actual risks are less than ½ the level of concern.

#### 4.3.6 Optimize the Design

As noted above, Phase IIB will collect groundwater data only from existing wells that are in suitable condition for sampling. Installation of additional wells and collection of additional groundwater data over space and time may be considered in the future if the results of the Phase IIB sampling effort indicate that the existing wells do not adequately characterize groundwater flow direction, gradient, and chemical concentrations over time.

#### 5.0 SAMPLING PROGRAM

All sampling of environmental media within OU3 described in this SAP will be performed by personnel who are properly trained in the field collection methods summarized in the OU3 Standard Operating Procedures (SOPs) provided in Attachment A and the Phase IIB experimental sampling design details presented below. A Health and Safety Plan (HASP) for the field sampling team will be provided by the field sampling contractor and reviewed by EPA prior to commencement of any field sampling activities.

#### 5.1 Ambient Air

#### 5.1.1 Experimental Design

The objective of the Phase IIB ambient air study is to characterize the current levels of LA release to air from the mine site. To this end, a total of eight stationary ambient air monitors have been established around the perimeter of the mined area. The locations of these monitoring stations are shown in Figure 5-1. Coordinates for these stations are provided in Attachment C. Stations A-4, A-5, A-6 and A-8 are at the same locations as were sampled in Phase I. Other station locations are new. As indicated, 5 of the 8 stations are located to the north and east of the mined area, since available meteorological data indicate that the predominant wind direction is to the north-east. However, 3 stations are located along the southern perimeter to capture any releases that may occur during wind reversals.

If the data from the initial ambient air monitoring phase suggest that there are frequent and significant releases from the mined area, then additional sampling stations may be added to identify the sources of the releases and to evaluate the dispersion of the releases.

#### 5.1.2 Sample Collection Protocol

Ambient air samples will be collected and equipment calibrated using the same SOP as used in the Phase I ambient air sampling program (SOP AMB-LIBBY-OU3, Rev. 1).

Because the objective of the sampling effort is to estimate long-term average concentration values, all ambient air samples will be collected using low-flow (2 L/min) stationary air monitors over a 5 day sampling period. In no event shall a sample be collected at a flow rate lower than 0.92 L/min, since the linear flow velocity would fall below 4 cm/sec, which is the minimum velocity specified by ISO 10312.

Samples will be collected using 25-mm diameter, 0.8 µm pore size MCE filter cassettes. All samples will be collected at a height approximately 6 feet above ground level.

Equipment shelters will be used to house the sampling pumps. The use of these shelters will protect the sampling equipment from adverse weather conditions that would otherwise interfere with the collection of long-term samples.

#### 5.1.3 Sample Collection Schedule

Sampling will begin immediately following approval of this SAP, and will continue through the fall of 2008 until weather conditions prohibit further sampling (estimated to be approximately mid-October). Samples will be collected from each station on a bi-weekly schedule (one sample per station every two weeks). Assuming that sampling begins in the first week of July, this corresponds to about 16 weeks (8 sampling rounds), or a total of about 64 samples.

All monitors will be checked periodically (at least once during each round of sample collection) to identify and correct any problems.

#### 5.1.4 Field QC Samples

Section 7.1.1 details field QC samples that will be collected for ambient air.

#### 5.1.5 Field Documentation

Field documentation procedures are described in Section 5.8 and OU3 SOP No. 9 (Rev 4). Field documentation associated with ambient air sampling will contain information of sufficient detail to fully describe:

- the location of each sampling station
- any problems encountered during sample collection (e.g., pump failures, weather damage to sampling stations, etc.)
- any other factors noted in the field that might influence the interpretation of the data

#### 5.2 Groundwater

#### 5.2.1 Experimental Design

As noted previously, groundwater sampling in Phase IIB will occur at existing wells in OU3 that are considered suitable for use.

Ten wells have been identified by MWH/Remedium in the vicinity of OU3. Table 5-1 summarizes information provided by MWH for each of these wells and Figure 5-2 shows the locations of the wells.

Based on the information available, five of the ten wells identified by MWH have been selected for sampling as part of the Phase IIB effort. These wells have been designated Wells A, C, D, E, and H. Well B is not included because it is plugged and abandoned. Well F is in poor condition, and it is believed Well H is a suitable surrogate. Wells G, I, and J are not included because no information is presently available on the type or condition of these wells.

#### 5.2.2 Sampling Methods and Procedures

At each well scheduled for sampling, field personnel will photograph and record a brief description of the well condition and location in relation to permanent landmarks. The Global Positioning System (GPS) coordinates of each well will be recorded in accordance with OU3 SOP 11 (Rev. 1), GPS Data Collection. Water levels will be measured and groundwater samples collected for analyses in accordance with OU3 SOP 13 (Rev. 0) and OU3 SOP 6 (Rev. 0).

Although sampling will not occur at wells G, I, and J during Phase IIB, these wells will be similarly inspected and characterized so that decisions can be made regarding their suitability for potential use in the future.

Since the wells scheduled for sampling have not been used for an extended period of time, all wells shall be re-developed prior to sampling to ensure that a representative groundwater sample is collected. SOP Libby Well Redevelop (Rev. 0) provides a detailed procedure for re-developing a monitoring well.

OU3 SOP Nos. 6 and 13 provide detailed procedures for groundwater sample collection and for measuring groundwater levels and purging wells prior to sampling. Water level will be measured before the well is purged, and groundwater samples will be collected after purging is complete.

All sampling and field measurement equipment that is used at more than one sample location must be decontaminated following each use. Appropriate equipment decontamination procedures are provided in OU3 SOP No. 7 (Rev. 0). Purge water and groundwater and other investigation derived waste shall be managed in accordance with OU3 SOP 12 (Rev. 0).

#### 5.2.3 Sample Containers and Preservation

Details on the specific analyses that will be performed for groundwater samples are discussed in Section 6. In accord with these analytical requirements, several different samples of water are required per well per sampling round. These requirements are summarized in Table 5-2.

The analytical laboratories will supply all sample containers utilized for this investigation, with preservative pre-added. If field personnel observe any cracked or dirty containers, or if the

appropriate preservative is missing in the sample bottles, those containers will be discarded and the laboratory will be notified of the problem to prevent its re-occurrence.

#### 5.2.4 Field QC Samples for Groundwater

Section 7.1.2 details field QC samples that will be collected for groundwater.

#### 5.2.5 Field Data Measurements

Measurements of pH, specific conductance, turbidity, dissolved oxygen, and temperature will be made in the field using individual or multiple probe electronic meters. Field parameter measurement and calibration protocols will be performed according to equipment manufacturer's specifications and OU3 SOP No. 10 (Rev. 1). Well-depth and depth-to-water measurements will be made in accordance with OU3 SOP No. 13 (Rev. 0) at each well where such measurements are possible.

Field measurement values are generally reported directly in the units of final use in the field notebook and data sheets without need for additional calculations (e.g., pH, temperature, and conductivity measurements). The field data will be reviewed daily by the field supervisor to identify anomalous data and transcriptional and/or computational errors. Corrective actions will be initiated as appropriate; these actions may consist of re-measuring a particular parameter, collecting a new sample, or other applicable corrective action measures.

#### 5.2.6 Field Documentation

Field documentation procedures are described in Section 5.8 and OU3 SOP No. 9 (Rev. 4). Field documentation associated with groundwater sampling will also contain information of sufficient detail to fully describe:

- well condition,
- total depth of well,
- depth to water,
- volume of water purged,
- final depth to water after sampling, and
- associated field measurements and field measurement methods.

#### 5.3 Sample Documentation and Identification

Data regarding each sample collected will be documented in accord with OU3 SOP No. 9 (Rev. 4) using Libby-specific field sample data sheets (FSDS). Any special circumstances that influence sample collection or result in deviations from sampling SOPs will be documented in a field log book.

At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number. The sample IDs for all samples collected as part of Phase II sampling activities will have a prefix of "P2" (e.g., P2-12345). Information on whether the sample is representative of a field sample or a field-based quality control (QC) sample (e.g., field blank, field split) will be documented on the FSDS, but this information will not be included on the chain-of-custody to make certain that the sample type is unknown to the analytical laboratory.

Each field sampling team will maintain a field log book. The log book shall record all potentially relevant information on sampling activities and conditions that are not otherwise captured on the FSDS forms. Examples of the type of information to be captured in the field log include:

- Names of team members
- · Current and previous weather conditions
- Field sketches
- Physical description of the location relative to permanent landmarks
- Number and type of samples collected
- Any special circumstances that influenced sample collection

As necessary for sample collection and location documentation, photographs will be taken using a digital camera. GPS coordinates will be recorded for all sampling locations on the FSDS form. A flag, stake or pole identifying the sampling station will be placed at or near the sampling station for future identification of the location.

#### 5.4 Sample Chain-of-Custody and Shipment

Field sample custody and documentation will follow the requirements described in OU3 SOP No. 9 (Rev. 4). Sample packaging and shipping will follow the requirements described in OU3 SOP No. 8 (Rev. 0).

A chain-of-custody form specific to the OU3 sampling shall accompany every shipment of samples to the analytical laboratory. The purposes of the chain-of-custody form are: a) to establish the documentation necessary to trace possession from the time of collection to final disposal, and b) to identify the type of analysis requested. All corrections to the chain-of-custody record will be initialed and dated by the person making the corrections. Each chain-of-custody form will include signatures of the appropriate individuals indicated on the form. The originals will accompany the samples to the laboratory and copies documenting each custody change will be recorded and kept on file. One copy of the chain-of-custody will be kept by field personnel.

All required paper work, including sample container labels, chain-of-custody forms, custody seals and shipping forms will be fully completed in ink (or printed from a computer) prior to shipping of the samples to the laboratory. Shipping to the appropriate laboratory from the field or sample storage will occur through overnight delivery.

All samples that may require special handling by laboratory personnel to prevent potential exposure to LA or other hazardous substances will be clearly labeled.

Upon receipt, the samples will be given to the laboratory sample custodian. The shipping containers will be opened and the contents inspected. Chain-of custody forms will be reviewed for completeness and samples will be logged and assigned a unique laboratory sample number. Any discrepancies or abnormalities in samples will be noted and the EPA Project Manager or the appropriate delegate will be promptly notified. The EPA Project Manager shall be notified by telephone at (303) 312-6579 or email at lavelle.bonita@epa.gov.

Chain-of-custody will be maintained until final disposition of the samples by the laboratory and acceptance of analytical results.

#### 6.0 LABORATORY ANALYSIS REQUIREMENTS

#### 6.1 Analysis of LA in Air or Water

#### 6.1.1 Laboratory Qualifications

All laboratories that analyze samples of air or water for asbestos as part of this project must participate in and have satisfied the certification requirements in the last two proficiency examinations from the National Institute of Standards and Technology/National Voluntary Laboratory Accreditation Program (NVLAP). Laboratories must also have demonstrated proficiency by successful analysis of Libby-specific performance evaluation samples and/or standard reference materials, and must participate in the on-going laboratory training program developed by the Libby laboratory team.

#### 6.1.2 Analytical Method

All samples of air or water collected during Phase IIB sampling will be submitted for asbestos analysis using transmission electron microscopy (TEM) in accord with the International Organization for Standardization (ISO) 10312 method (ISO 1995) counting protocols, with all applicable Libby site-specific laboratory modifications, including the most recent versions of modifications LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000053, LB-000066, and LB-000085 (see Attachment B). All amphibole structures (including not only LA but all other asbestos types as well) that have appropriate Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra, and having length greater than or equal to 0.5 um and an aspect ratio (length:width) ≥ 3:1, will be recorded on the Libby site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheets. Data recording for chrysotile, if observed, is not required.

All groundwater samples collected will be prepared for analysis by filtering an aliquot of the samples (generally about 100 mL) through a 47 mm mixed cellulose acetate (MCE) filter with pore size of 0.2 um, using a backing filter with pore size of 5 um.

#### 6.1.3 Stopping Rules

Field Samples and Field Duplicates

For field samples and field duplicates, the initial stopping rules are as follows:

Count the sample until one of the following is achieved:

- The target sensitivity is achieved
- 25 LA structures are observed
- 50 grid openings are evaluated

When one of these goals is achieved, complete the final grid opening and stop. These stopping rules may be revised as data become available on the levels of LA and other particles that are present in the field samples.

#### Field Blanks and Lot Blanks

For field blanks and lot blanks, count 10 grid openings and stop.

#### Estimated Grid Opening Requirements

As discussed previously, the target analytical sensitivity for ambient air samples is 0.001 cc<sup>-1</sup>. Assuming that typical sample volumes for ambient air samples will be about 10,000 L and that indirect preparations are not necessary, it is expected that an analytical sensitivity of 0.001 cc<sup>-1</sup> can be achieved by counting about 5 TEM grid openings (GOs).

The target analytical sensitivity for LA in groundwater is 50,000 f/L (0.05 million fibers per liter, abbreviated as MFL). This sensitivity can be achieved by filtering 100 mL of water and counting about 25 grid openings (GOs), assuming that filter overloading does not occur.

#### 6.1.4 Holding Times

There are no holding times for the analysis of LA. However, for analysis of water samples, preparation of filters should be performed within 1-2 days of sample collection in order to minimize the growth of algae or bacteria that may interfere with the TEM analysis.

## 6.2 Analytical Methods for Other (Non-Asbestos) Analytes in Groundwater

#### 6.2.1 Analyte List for Groundwater

The target analyte list for groundwater in Phase IIB includes not only LA but also other chemical classes that might have been used at the site or that might have been released to the environment due to mining activities. This includes Target Analyte List (TAL) metals/metalloids, major ions, volatile and extractable petroleum hydrocarbons (VPH and EPH), gross alpha/gross beta, and cyanide. This list of non-asbestos analytes and the associated analytical methods are presented in Table 6-1.

All groundwater samples will be analyzed for total EPH using method SW8015M. If the total EPH concentration is greater than 300 ug/L, the water sample will be analyzed for specific EPH

compounds (C9-C18 aliphatics, C19-C36 aliphatics, and C11-C22 aromatics) and PAHs<sup>1</sup> using method MADEP-EPH-04-1.1.

The laboratory performing chemical analyses is required to follow procedures for each referenced method in accordance with the method protocols in the original source documents. All method-specific quality control measures, such as external and internal standard calibration procedures, instrument performance verifications, and quantitation using method of standard additions, specified within any referenced EPA method number will be performed.

#### 6.2.2 Target Detection Limits

The analytical methods identified in Table 6-1 were selected to provide quantitative results at or below state/federal drinking water standards and tap water risk-based concentrations (RBCs) for residents (see Attachment B in USEPA 2007 *Phase I Sampling and Analysis Plan for Operable Unit 3*).

#### 6.2.3 Holding Times

A holding time is defined as the allowable time between sample collection and analysis and/or extraction recommended to ensure accuracy and representativeness of analysis results, based on the nature of the analyte of interest and chemical stability factors. The holding time is calculated from the date and time of sample collection to the time of sample preparation and/or analysis. Sample holding times are established to minimize chemical changes in a sample prior to analysis and/or extraction. Samples will be shipped to the laboratory as soon as possible after collection or processing. Table 5-2 defines method-specific analytical holding times for groundwater analyses.

#### 6.3 Instrument Calibration and Frequency

All laboratory instruments used in the analysis of samples generated during this project must be calibrated by the laboratory in accordance with the requirements of the instrument manufacturer and the requirements specified in the relevant analytical method. Calibration records will be kept in logbooks for all instruments. It is the responsibility of the Laboratory Quality Assurance (QA) Officer to assure that calibration data is properly logged in the logbooks for each analysis.

#### 6.4 Laboratory Custody Procedures and Documentation

The laboratories will implement the following procedures:

A sample custodian will be designated.

<sup>&</sup>lt;sup>1</sup> PAH analyte list: acenaphthene, anthracene, benz(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, and pyrene

- Upon receipt at the laboratory, each sample shipment will be inspected to assess the condition of the shipping container and the individual samples.
- Enclosed chain-of-custody records will be cross-referenced with all the samples in the shipment. These records will be signed by the sample custodian and placed in the project file.
- Sample storage will be secured (in the appropriate environment, i.e., refrigerated, dry, etc.), sample storage records and intra-laboratory sample custody records will be maintained, and sample disposal and disposal date will be properly documented.
- Internal chain-of-custody procedures will be followed by assigning a unique laboratory number to each sample on receipt; this number identifies the sample through all further handling;
- Internal logbooks and records will maintain the chain-of-custody throughout sample preparation and analysis, and data reporting will be kept in the project files.
- The original chain-of-custody record will be returned to the Project QA Officer with the resulting data report from the laboratory.

It is the laboratory's responsibility to maintain internal logbooks and records throughout sample preparation, analysis, and data reporting.

# 6.5 Laboratory Health and Safety

All laboratories analyzing samples from OU3 must be properly trained in the safe handling, storage and disposal of samples that may contain LA and other potentially hazardous materials.

### 6.6 Documentation and Records

Data reports will be submitted to EPA in accordance with the procedures described in Section 6.8. Data reports shall include a case narrative that briefly describes the number of samples, the analyses, and any analytical difficulties or QA/QC issues associated with the submitted samples. The data report will also include signed chain-of-custody forms, analytical data summary report pages, and a summary of laboratory QC sample results and raw data, where applicable. Raw data are to consist of instrument preparation and calibration logs, instrument printouts of field sample results, laboratory QC sample results, calibration and maintenance records, chain-of-custody check in and tracking, raw data count sheets, spectra, micrographic photos, and diffraction patterns.

# 6.7 Sample Archival and Final Disposition

Unused samples and containers will be maintained in storage at the laboratory for a minimum of 90 days following completion of the analysis, unless otherwise directed by EPA. After 90 days or approval from EPA for disposal, the laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and packing materials in accordance

with sound environmental practice, based on the sample analytical results. The laboratory will maintain proper records of waste disposal methods, and will have disposal company contracts on file for inspection.

#### 6.8 Data Deliverables

Asbestos data generated during this project will be entered into Libby-specific EDD spreadsheets by appropriately trained data entry staff. The data will include all relevant field information regarding each environmental sample collected, as well as the analytical results provided by the laboratory. Analytical results will include the structure-specific data for all TEM analyses. All data entry will be reviewed and validated for accuracy by the laboratory data entry manager or appointed delegate.

Non-asbestos data generated for this project will be transmitted via an EDD spreadsheet. The specific structure and format of this spreadsheet will be specified by the project data manager and will be provided to the laboratory for data submittal. All data entry will be reviewed and validated for accuracy by the laboratory data entry manager or appointed delegate.

All asbestos and non-asbestos EDDs will be submitted to EPA technical contractors (SRC) electronically. Whenever possible, data files should be transmitted by e-mail to the following address:

LibbyOU3@syrres.com

When files are too large to transmit by e-mail, they should be provided on compact disk to the following address:

Lynn Woodbury Syracuse Research Corporation 999 18<sup>th</sup> Street, Suite 1975 Denver CO 80202 (303) 357-3127

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by the Project Manager. At the termination of Phase II, all original data records will be provided to the EPA Project Manager in a format specified by EPA for incorporation into the OU3 project files.

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## 7.0 QUALITY CONTROL

Quality Control (QC) is a component of the QAPP, and consists of the collection of data that allow a quantitative evaluation of the accuracy and precision of the field data collected during the project. QC samples that will be collected during this project include both field-based and laboratory-based QC samples.

# 7.1 Field-Based Quality Control Samples

Field-based QC samples are samples which are prepared in the field and submitted to the laboratory in a blind fashion. That is, the laboratory is not aware the sample is a QC sample, and should treat the sample in the same way as a field sample. In general, there are three types of field QC sample: blanks, field splits/duplicates, and performance evaluation (PE) samples. A summary of the field-based QC samples can be found in Table 7-1. The following sections describe the field QC samples that will be collected for ambient air and for groundwater.

## 7.1.1 Field QC Samples for Air

## Lot Blanks

Before any air cassettes may be used for asbestos sampling, the lot must be determined to be asbestos free. This will be accomplished by selecting 2 lot blanks at random from the group of cassettes to be used for collection of ambient air samples. Each lot blank will be submitted for TEM analysis using ISO 10312 counting protocols as modified by Libby-specific laboratory modifications, including project specific modifications LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000053, LB-000066, and LB-000085. Once the lot is confirmed to be asbestos free (i.e., both lot blanks are non-detect after evaluation of 10 grid openings), that lot may be placed into use for sampling.

### Field Blanks

A field blank for air shall be prepared by removing the sampling cassette from the box, opening the cassette to the air in the area where the investigative samples will be taken, then closing the cassette and packaging for shipment and analysis. Field blanks for air will be collected at a rate of 1 per sampling round (1 per 2 weeks). If contamination is detected in any of these field blanks, then the frequency of field blanks may be increased.

# Field Duplicates

A field duplicate is a field sample that is collected at the same place and time as an original field sample. Field duplicates will be collected for ambient air at a rate of 1 per 10 field samples. The

specific stations at which field duplicates will be collected will be determined in the field based on sampling conditions.

# 7.1.2 Field QC Samples for Groundwater

### Field Blank

A field blank for water shall be prepared by placing an appropriate volume of analyte-free reagent water (e.g., ASTM Type II) into a sample collection container in the field. Field blanks for water will be collected at a rate of at least 10% (1 field blank per 10 field samples, or 1 per sample batch, whichever is greater).

# Trip Blanks

The trip blank for water is used to indicate potential contamination by volatile chemicals during sample shipping and handling. A trip blank consists of analyte-free laboratory reagent water which accompanies the empty sample bottles to the field and is placed in each cooler containing samples scheduled for volatile chemical (VPH or EPH) analysis. The trip blank is not opened until analysis in the laboratory with the corresponding site samples.

During Phase IIB sampling, one trip blank per cooler will be prepared to accompany aqueous samples when they are shipped to the laboratory for VPH or EPH analysis.

### Field Splits

A field split is a sample that is prepared by thoroughly homogenizing a field sample, dividing the homogenized sample into two parts, and analyzing each independently. A comparison of field split samples is a measure of the precision of the sample preparation and analysis methods. Field splits for water will be collected at a rate of approximately 10% (1 field split per 10 field samples) for each analysis.

#### Performance Evaluation Samples

Performance Evaluation (PE) samples are samples of a matrix that contain a known and certified level of a contaminant. The results of PE sample analysis help evaluate analytical accuracy. PE samples for water are available through the EPA Quality Assurance Technical Support (QATS) program. A total of 4 water PE samples containing a range of inorganic and organic analytes will be submitted to the analytical laboratories for preparation and analysis with the field samples.

## **Equipment Rinsate Blanks**

Equipment rinsate blanks determine if decontamination procedures of field equipment are adequate to prevent cross-contamination of samples during sample collection. An equipment rinsate blank is prepared by rinsing decontaminated field equipment with analyte-free reagent water. Equipment rinsate blanks will be collected at a rate of 1 per sampling team per day. If field equipment is not re-used between sampling locations (i.e., dedicated equipment is used or equipment is disposable and decontamination is not necessary), equipment rinsate blanks will not be collected.

# 7.2 Laboratory-Based Quality Control Samples

Laboratory-based QC samples are samples and analyses performed in the laboratory to establish the reliably of the data being generated. Laboratory-based QC samples for LA and other analytes are presented below.

## 7.2.1 Laboratory-Based QC for LA

The QC requirements for TEM analyses of air and water samples at the Libby site are patterned after the requirements set forth by NVLAP. There are three types of laboratory-based QC analyses that are performed for TEM. Each of these is described in more detail below.

Lab Blank - This is an analysis of a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples.

Recounts - A recount is an analysis where TEM grid openings are re-examined after the initial examination. The type of recount depends upon who is performing the re-examination. A Recount Same (RS) describes a re-examination by the same microscopist who performed the initial examination. A Recount Different (RD) describes a re-examination by a different microscopist within the same laboratory than who performed the initial examination. An Interlab (IL) describes a re-examination by a different microscopist from a different laboratory.

Repreparation - A repreparation is an analysis of a TEM grid that is prepared from a new aliquot of the same field sample as was used to prepare the original grid. Typically, this is done within the same lab as did the original analysis, but a different lab may also prepare grids from a new piece of filter.

As described the most recent Libby-specific Laboratory Modification #29 (LB-000029 in Attachment B), lab blanks will be performed at a frequency of 4%, recounts will be performed at a frequency of 5%, and repreparations will be performed at a frequency of 1%. LB-000029

summarizes the project-specific acceptance criteria for TEM QC analyses for all participating laboratories.

# 7.2.2 Laboratory-Based Quality Control Samples for Non-Asbestos Analyses

The following subsections describe laboratory-based quality control measures used to assess and document the quality of analytical results for non-asbestos parameters. Laboratory QC sample analysis frequencies and control limits used by contracted laboratories will be in accordance with referenced analytical method protocols, and the QC analyses and results will be documented and reported to EPA by the selected laboratory.

Table 7-2 summarizes all laboratory quality control measures, control limits, and corrective actions for this project, by analysis method. All laboratory QC data will be reported with results of associated sample analyses to allow for comparison of QC results to the QC criteria specified for this project.

#### Method Blank

Method blanks are designed to measure laboratory-introduced contamination of environmental samples. Method blanks verify that method interferences caused by airborne contaminants, solvents, reagents, glassware, or other sample processing hardware are known and minimized. The blank will be ASTM Type II water (or equivalent) for water samples. The method/reagent blank is processed through all procedures, materials, and lab-ware used for sample preparation and analysis.

The frequency for method blank preparation and analysis is a minimum of one per twenty field samples or per analytical batch, whichever is most frequent. An analytical batch is defined as samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch are to be of similar composition or matrix.

Acceptance criteria and corrective action for out-of-control method blanks are provided in Table 7-2.

# Laboratory Control Samples

Laboratory control samples (LCSs) are designed to check the accuracy of an analytical procedure by measuring a known concentration of an analyte of interest. LCS samples are prepared by spiking clean, laboratory-simulated matrices (reagent-free water or purified solid matrix) with representative analytes at known concentrations that are approximately 10 times greater than the method's quantitation limits. These spiked samples are then subjected to the same preparation

and analytical procedures as associated environmental samples. A LCS will be analyzed with every analytical batch, and the measured concentrations will be compared to the known, or spiked, concentrations of the LCS to compute a percent recovery value.

LCSs will be analyzed at a minimum frequency of one per every 20 samples or one per analytical batch of no more than 20 samples. Control limits for laboratory control samples are listed on Table 7-2. Failure of the LCS to meet recovery criteria requires corrective action before any further analyses can continue.

For some methods, a duplicate of the LCS is also analyzed with each analytical batch and the difference between the LCS and the LCS Duplicate (LCSD) indicates the precision of laboratory sample preparation and analysis methods at a known concentration level. Control limits for precision measured by the RPD of LCS/LCSD results are listed in Table 7-2. When LCSD samples are analyzed, the minimum frequency of analysis is one per every 20 samples.

# Matrix Spikes/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) samples are designed to evaluate the effect of the sample matrix on analytical data, by measuring precision and accuracy from a known concentration of a target analyte that has been added to a particular sample matrix. MS/MSD samples are prepared by spiking environmental field samples with a standard solution containing known concentrations of representative target analytes. The MS/MSD sample pair is prepared from three volumes of an environmental sample. Two portions of the sample (the MS and the MSD) are spiked with the standard solution. The remaining volume is not spiked. The spiked samples are analyzed, and the percent recovery (PR) and relative percent difference (RPD) between the results of the MS analysis and the MSD analysis are calculated. The unaltered sample volume is analyzed as an ordinary environmental sample.

Sampling personnel will identify for the laboratory which samples are to be used for MS/MSD preparation. Field blanks and field duplicates are not used as MS/MSDs. Typically, additional sample volume will be required to prepare the MS and MSD, especially for analyses of water samples for organic compounds. MS/MSDs will be analyzed at a minimum frequency of one per every 20 samples.

Background and interferences that have an effect on the actual sample analyte will have a similar effect on the spike. The calculated percent recovery of the matrix spike is considered to be a measure of the relative accuracy of the total analytical method, i.e., sample preparation and analysis. The matrix spike is also a measure of the effect of the sample matrix on the ability of the methodology to detect specific analytes. Acceptance criteria and corrective action procedures for out-of-control matrix spike results are listed in Table 7-2.

## Surrogate Spike Analyses

Surrogate spike analyses are used to determine the efficiency of target analyte recovery during sample preparation and analysis. A surrogate spike is prepared by adding a known amount of surrogate compound to an environmental sample before extraction. The surrogate compound is selected to exhibit an analytical response that is similar to the response displayed by a target compound during sample analysis. The accuracy of the analytical method is measured using the calculated percent recovery of the spiking compound. Poor reproducibility and percent recovery during surrogate spike analyses may indicate sample matrix effects.

Surrogate compounds are not added to inorganic analyses; however, surrogates are required for most organic analyses. Both environmental and QC samples are spiked with surrogate compounds. Surrogate spike recoveries are acceptable if the results of a surrogate spike fall within the control limits established by laboratory QC protocol. Acceptance criteria and corrective action procedures for out-of-control surrogate spike results are listed in Table 7-2.

Frequencies for surrogate spike analyses will be consistent with the referenced method protocols.

#### Internal Standards

Internal Standards (ISs) are compounds of known concentrations used to quantitate the concentrations of target detections in field and QC samples. ISs are added to all samples after sample extraction or preparation. Because of this, ISs provide for the accurate quantitation of target detections by allowing for the effects of sample loss through extraction, purging, and/or matrix effects. ISs are used for any method requiring an IS calibration. Corrective action is required when ISs are out of control. Acceptance criteria and corrective action procedures for out-of-control internal standard spike results are listed in Table 7-2.

# 7.3 Instrument Calibration and Frequency

Analytical instruments will be calibrated in accordance with the referenced analytical methods. All target analytes that are reported to EPA will be present in the initial and continuing calibrations, and these calibrations must meet the acceptance criteria specified in referenced methods. Records of standard preparation and instrument calibration will be maintained by the contract laboratory. Records will unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards will be traceable to standard materials.

Analyte concentrations are determined with either calibration curves (linear regression) or response factors (RFs). All correlation coefficients for linear regression calibration curves or relative standard deviation (RSD) of RFs to determine linearity must meet the acceptability criteria specified within the method. For GC/MS methods, the average RF from the initial five-

point calibration will be used to determine analyte concentrations. The continuing calibration curve will not be used to update the RFs from the initial five-point calibration. GC/MS methods also will meet all instrument performance and/or tuning criteria as specified by the methods.

#### **Initial Calibration Verification**

Initial calibration curves must be verified using a standard made from a source independent of the one used to make the initial calibration standards. All target compounds must be included within the initial calibration verification (ICV), typically at a concentration around the midpoint of the calibration curve. Control limits and corrective action procedures for out-of-control initial calibration verification results are listed in Table 7-2.

## Continuing Calibration and Verification

Initial calibration curves must be verified daily prior to sample analysis. All target compounds must be included, typically at a concentration around the midpoint of the calibration curve. Continuing calibration verifications (CCVs) are check samples required at frequencies specified in each analytical method, typically at the beginning and end of each analytical sequence and after every ten samples analyzed (as specified in each analytical method). Control limits and corrective action procedures for out-of-control CCV results are listed Table 7-2.

Calibration procedures for a specific laboratory instrument will consist of initial calibration (3-or 5-points), initial calibration verification (ICV) and continuing calibration verification (CCV). Calibration protocols included in method references, including calibration frequencies, conditions, and acceptance criteria, will be followed.

### 7.4 Quality Assurance Objectives For Measurement Data

This section identifies specific objectives for precision, accuracy, representativeness, completeness, and comparability of measurement data collected to support the Phase II data quality objectives.

#### 7.4.1 Precision

Precision is defined as the agreement between a set of replicate measurements without assumption or knowledge of the true value. Agreement is expressed as either the relative percent difference (RPD) for duplicate measurements, or the range and standard deviation for larger numbers of replicates. Precision will be assessed through the calculation of the relative percent difference (RPD) for two replicate samples. RPD is calculated according to the following formula:

$$RPD = \frac{(S-D)}{(S+D)/2} \cdot 100$$

where:

S = Original sample value

D = Duplicate sample value

Field precision is assessed through the collection and measurement of field duplicates. The variability between field duplicates reflect the combined variation in concentration between nearby samples and the variation due to measurement error. Because the variability between field duplicates is random and may be either small or large, no quantitative requirement for the agreement of field duplicates is established for this project.

Precision in the laboratory is assessed through calculation of RPDs for duplicate analyses or relative standard deviations (RSDs) for three or more replicate analyses of the same sample. RPD of 25% for water field duplicates will be used as an advisory limit for analytes detected in both the original sample and its field duplicate at concentrations greater than 5 times the reported quantitation limit.

Differences greater than these advisory limits will be noted for data users through the data validation process.

# 7.4.2 Accuracy

Accuracy is a measure of the agreement between a measurement and the "true" value. The accuracy of a measurement may be affected by errors introduced by field contamination, sample preparation and handling, and sample analysis. The accuracy of an analytical method is generally assessed by analyses of samples with known concentration levels, including field calibration standards (for field based measurements), laboratory control samples, MS/MSD samples, and PE samples.

The accuracy required for data usability depends on a number of factors. In general, good accuracy is most important for samples whose concentration values are close to the level of concern, and a somewhat lesser level of accuracy may be acceptable for samples whose concentrations are either well below or well above a level of concern. Based on this, the goal of Phase II is to achieve an analytical accuracy of  $\pm 25\%$  for analytes that are within a factor of 10 of initial estimates of the level of concern, and  $\pm 50\%$  for samples either 10-fold above or 10-fold below initial estimates of the level of concern.

# 7.4.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent characteristics of a population, parameter variations at a sampling point, or an environmental condition.

Representativeness of field measurements is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the SAP and SOPs are followed. The Phase II sampling activities are designed to provide data that are representative of conditions at specific locations and times of sample collection.

# 7.4.4 Completeness

Data are considered complete when a prescribed percentage of the total intended measurements and samples are obtained. Analytical completeness is defined as the percentage of valid analytical results requested.

Field completeness is a measure of the amount of valid measurement data collected for the project. The target completeness objective for field measurements collected for this sampling program is 95% or more.

Laboratory completeness is a measure of the amount of valid laboratory-measurement data obtained for the project. For this sampling program, a minimum of 90% of the planned collection of individual samples for quantification must be obtained to achieve a satisfactory level of data completeness.

# 7.4.5 Comparability

Data are comparable if collection techniques, measurement procedures, methods, and reporting units are equivalent for the samples within a sample set. These criteria allow comparison of data from different sources. Comparable data will be obtained by specifying standard units for physical measurements and standard procedures for sample collection, processing, and analysis.

The criteria for field comparability will be to ensure and document that the sampling designs are properly implemented and the sampling procedures are consistently followed for the duration of the Phase II data collection program. Each sampling task will utilize standardized procedures for sample collection and field measurements, as specified in Section 5 of this plan.

The criteria for laboratory data comparability will be to ensure that the laboratory results generated during Phase II will be comparable to laboratory data collected for future environmental investigations at OU3 and comparable to the asbestos data already collected by EPA in the vicinity of OU3. This goal will be achieved through utilization of standard EPA Test Methods and site-specific asbestos analysis methods for sample analyses and adherence to quality assurance/quality control and analytical procedures specified for the OU3 RI.

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## 8.0 DATA MANAGEMENT

# 8.1 Data Applications

All data generated as part of the Phase II sampling event will be maintained in an OU3-specific Microsoft<sup>®</sup> Access database. This will be a relational database with tables designed to store information on station location, sample collection details, preparation and analysis details, and analytical results. Results will include asbestos data (including detailed structure attributes for TEM analyses) and non-asbestos chemical data (e.g., metals).

As needed, EPA staff and designated contractors will develop tabular and graphical data summaries, perform statistical analyses, and generate maps using commercially-available applications such as Microsoft® Access and Excel and ArcGIS®.

# 8.2 Roles and Responsibilities for Data Flow

### 8.2.1 Field Personnel

W.R. Grace Contractors will perform all Phase II sample collection in accordance with the project-specific sampling plan and SOPs presented above. In the field, sample details will be documented on hard copy media-specific FSDS forms and in field log books (see Section 5.3). chain-of-custody information will be documented on hard copy forms (see Section 5.4). FSDS and chain-of-custody information will be manually entered into a field-specific<sup>2</sup> OU3 database using electronic data entry forms. Use of electronic data entry forms ensures the accuracy of data entry and helps maintain data integrity. For example, data entry forms utilize drop-down menus and check boxes whenever possible. These features allow the data entry personnel to select from a set of standard inputs, thereby preventing duplication and transcription errors and limiting the number of available selections (e.g., media types). In addition, entry into a database allows for the incorporation of data entry checks. For example, the database will allow a unique sample ID to only be entered once, thus ensuring that duplicate records cannot be created.

Entry of FSDS forms and chain-of-custody information will be completed weekly, or more frequently as conditions permit. Copies of all FSDS forms, chain-of-custody forms, and field log books will be scanned and posted in portable document format (PDF) to a project-specific file transfer protocol (FTP) site weekly. This FTP site will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned FSDS forms, chain-of-custody forms, and field log books will include the sample date in the format YYYYMMDD to facilitate document organization (e.g., FSDS\_20070831.pdf). Electronic copies of all digital photographs will also be posted weekly to

<sup>&</sup>lt;sup>2</sup> The field-specific OU3 database is a simplified version of the master OU3 database. This simplified database includes only the station and sample recording and tracking tables, as well as the FSDS and chain-of-custody data entry forms.

the project-specific FTP site. File names for digital photographs will include the station identifier, the sample date, and photograph identifier (e.g., ST-1 20070831 12459.tif).

After FSDS data entry is completed, a copy of the field-specific OU3 database will be posted to the project-specific FTP weekly, or more frequently as conditions permit. The field-specific OU3 database posted to the FTP site will include the post date in the file name (e.g., FieldOU3DB\_20070831.mdb).

## 8.2.2 Laboratory Personnel

Each of the laboratories performing analyses for the Phase II sampling event are required to utilize all applicable Libby-specific Microsoft<sup>®</sup> Excel spreadsheets for data recording and electronic submittals (see Section 6.8). Upon completion of the appropriate analyses, EDDs will be transmitted via email to a designated email distribution list within the appropriate turn around time. Hard copies of all analytical laboratory data packages will be scanned and posted as a PDF to the project-specific FTP site. File names for scanned analytical laboratory data packages will include the laboratory name and the job number to facilitate document organization (e.g., LabX\_12365-A.pdf).

#### 8.2.3 Database Administrators

Day-to-day operations of the master OU3 database will be under the control of EPA contractors. The primary database administrator will be responsible for sample tracking, uploading new data, performing error checks, and making any necessary data corrections. New records will be added to the master OU3 database within an appropriate time period of FSDS and/or EDD receipt.

Incremental backups of the master OU3 database will be performed daily Monday through Thursday, and a full backup will be performed each Friday. The full backup tapes will be stored off-site for 30 days. After 30 days, the tape will be placed back into the tape library to be overwritten by another full backup.

Each Friday, a copy of the master OU3 database will be posted to a project-specific FTP site to allow timely access to results by data users. The master OU3 database posted to the FTP site will include the post date in the file name (e.g., MasterOU3DB\_20070831.mdb).

## 8.3 Data Storage

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by the EPA Project Manager. At the termination of this project, all original data records will be provided to the EPA Project Manager in a format specified by EPA for incorporation into the site project files.

#### 9.0 ASSESSMENT AND OVERSIGHT

Assessments and oversight reports to management are necessary to ensure that procedures are followed as required and that deviations from procedures are documented. These reports also serve to keep management current on field activities. Assessment, oversight reports, and response actions are discussed below.

#### 9.1 Assessments

### 9.1.1 Field Oversight

All individuals who collect samples during field activities will be provided a copy of this SAP and will be required to participate in a pre-sampling readiness review meeting to ensure that methods and procedures called for in this SAP and associated SOPs are understood and that all necessary equipment is on hand. EPA may perform random and unannounced field audits of field sampling collection activities, as may be deemed necessary.

#### 9.1.2 Laboratory Oversight

All laboratories selected for analysis of samples for asbestos will be part of the Libby analytical team. These laboratories have all demonstrated experience and expertise in analysis of LA in environmental media, and all are part of an on-going site-specific quality assurance program designed to ensure accuracy and consistency between laboratories. These laboratories are audited by EPA and NVLAP on a regular basis. Additional laboratory audits may be conducted upon request from the EPA, as may be needed.

# 9.2 Response Actions

If any inconsistencies or errors in field or laboratory methods and procedures are identified, response actions will be implemented on a case-by-case basis to correct quality problems. All response actions will be documented in a memo to the EPA RPM for OU3 at the following address:

Bonita Lavelle
U.S. EPA Region 8
1595 Wynkoop Street
Denver, CO 80202-1129
E-mail: lavelle.bonita@epa.gov

Any problems that cannot be corrected quickly through routine procedures may require implementation of a corrective action request (CAR) form.

# 9.3 Reports to Management

Field and analytical staff will promptly communicate any difficulties or problems in implementation of the SAP to EPA, and may recommend changes as needed. If any revisions to this SAP are needed, the EPA RPM will approve these revisions before implementation by field or analytical staff.

#### 10.0 DATA VALIDATION AND USABILITY

## 10.1 Data Validation and Verification Requirements

Data validation consists of examining the sample data package(s) against pre-determined standardized requirements. The validator may examine, as appropriate, the reported results, QC summaries, case narratives, chain-of-custody information, raw data, initial and continuing instrument calibration, and other reported information to determine the accuracy and completeness of the data package. During this process, the validator will verify that the analytical methodologies were followed and QC requirements were met. The validator may recalculate selected analytical results to verify the accuracy of the reported information. Analytical results will then be qualified as necessary.

Data verification includes checking that results have been transferred correctly from laboratory data printouts to the laboratory report and to the EDD. Some of the data verification checks are performed as a function of built-in quality control checks in the Libby-specific data entry spreadsheets. Additional verifications of field and analytical results will be performed at a frequency of 10%. This initial rate may be revised as samples are analyzed and results evaluated. Data validation, review, and verifications must be performed on sample results before distribution to the public for review.

# 10.2 Reconciliation with Data Quality Objectives

Once all samples have been collected and analytical data has been generated, data will be evaluated to determine if DQOs were achieved. Evaluation of the Phase II data will include a qualitative and quantitative review of all QC samples and all deviations from sampling and analysis plans described in this report, along with conclusions regarding the reliability of the data for their intended use. Results of the data quality evaluation will in general be reported in the Baseline Human Health Risk Assessment, the Baseline Ecological Risk Assessment, and the final RI Report for OU3.

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Table 3-1. Ambient Air Field Sample Results

Station ID	Round	Index ID	Sensitivity 1/cc	LA Count	Conc s/cc
A-1	1	P1-00005	5.6E-04	0	0.0E+00
	2	P1-00017	5.6E-04	0	0.0E+00
	3	P1-00243	4.5E-04	0	0.0E+00
	4	P1-00277	5.6E-04	0	0.0E+00
	1	P1-00006	5.6E-04	0	0.0E+00
A-2	2	P1-00018	5.6E-04	0	0.0E+00
A-2	3	P1-00244	4.5E-04	0	0.0E+00
	4	P1-00278	5.6E-04	0	0.0E+00
	1	P1-00010	5.6E-04	0	0.0E+00
A-3	2	P1-00024	5.6E-04	0	0.0E+00
M-3	3	P1-00250	4.5E-04	0	0.0E+00
	4	P1-00284	5.6E-04	0	0.0E+00
	1	P1-00007	6.2E-04	0	0.0E+00
A-4	2	P1-00020	5.6E-04	0	0.0E+00
A-4	3	P1-00245	4.6E-04	0	0.0E+00
	4	P1-00279	5.6E-04	0	0.0E+00
	1	P1-00008	6.2E-04	0	0.0E+00
A-5	2	P1-00022	5.6E-04	0	0.0E+00
A-5	3	P1-00247	4.5E-04	0	0.0E+00
	4	P1-00281	5.6E-04	0	0.0E+00
	1	P1-00009	5.6E-04	0	0.0E+00
٨٥	2	P1-00023	5.6E-04	0	0.0E+00
A-6	3	P1-00249	4.5E-04	0	0.0E+00
	4	P1-00283	5.6E-04	0	0.0E+00
	1	P1-00001	5.6E-04	0	0.0E+00
A-7	2	P1-00015	5.6E-04	0	0.0E+00
	3	P1-00241	4.5E-04	0	0.0E+00
	4	P1-00275	5.6E-04	0	0.0E+00
	1	P1-00003	6.2E-04	0	0.0E+00
٨٥	2	P1-00016	8.0E-04	0	0.0E+00
A-8	3	P1-00242	4.5E-04	0	0.0E+00
	4	P1-00276	5.6E-04	0	0.0E+00

Round 1: 10/2/2007 - 10/7/2007 Round 2: 10/7/2007 - 10/12/2007 Round 3: 10/12/2007 - 10/17/2007 Round 4: 10/17/2007 - 10/22/2007

Table 5-2. Sample Containers, Preservation and Handling Requirements, and Holding Times for Groundwater Samples

Container Description	Analyses	Method	Preservation and Handling	Extraction/ Analysis Holding Times
250-mL plastic (pre-preserved with HNO <sub>3</sub> )	TAL Metals+Boron (Total)	6010B/6020 and EPA 200 series methods (a)	Cool 4°C; HNO <sub>3</sub> , pH<2	180 days
	Mercury	7470A/ EPA 245.1	Cool 4°C	28 days
250-mL plastic filtration container	TAL Metals+Boron (Dissolved), Hardness	6010B/6020 and EPA 200 series methods (a)	Cool 4°C; HNO <sub>3</sub> (preserve sample in field after filtering)	180 days
3 x 40-mL amber glass vial with Teflon-lined screw cap (pre- preserved with HCI)	Volatile Petroleum Hydrocarbons (VPH)	MA-DEP VPH modified	HCl to pH <2 Cool 4°C	14 days
2 x 1-L amber glass bottle with Teflon-lined screw cap (pre- preserved with H <sub>2</sub> SO <sub>4</sub> )	Extractable Petroleum Hydrocarbons (EPH)	SW8015M (e) MA-DEP EPH modified	H <sub>2</sub> SO <sub>4</sub> to pH <2, Cool 4°C	14 days/40 days
1-L plastic	Fluoride/Chloride/Sulfate	EPA 300.0	Cool 4°C	28 days
	Total Suspended Solids (TSS)	Standard Methods 2540D	Cool 4°C	7 days
	Nitrite	EPA 353.2	Cool 4°C	48 hours
	Total Dissolved Solids (TDS)	Standard Methods 2540C,D	Cool 4°C	7 days
	Alkalinity	Standard Methods 2320B	Cool 4°C	14 days
1-L plastic (pre-preserved with HNO <sub>3</sub> )	Radiochemistry (gross alpha and gross beta)	EPA 900.0	Cool 4°C; HNO <sub>3</sub>	None
500-mL plastic(pre-preserved with NaOH)	Cyanide	Kelada mod.	Cool 4°C; NaOH, pH>12	14 days
1 L HDPE container	Asbestos	ISO 10312 (d)	Cool 4°C	Filtered within 48 hours

<sup>(</sup>a) 200 series methods: 200.7, 200.8

<sup>(</sup>b) 2 additional 1-L amber glass containers will be needed for MS/MSD

<sup>(</sup>c) CLP analyte list

<sup>(</sup>d) with Libby-specific modifications

<sup>(</sup>e) SW8015M will be used as a screening method to determine if more detailed analysis by MA-DEP EPH is needed

TABLE 5-1. SUMMARY OF WELL INFORMATION PROVIDED BY MWH

MWH 2007 Well I.D.	Location Description	Well Diam. (in.)	Material	TD (ft. BTOC)	SWL (ft. BTOC)
A	"CCC Well" in Carney Creek drainage, upstream of pond below fine tailings	6	Steel	41.42	5.35
В	In grassy area downstream from Amphitheater, plugged and abandoned.	8	Steel	NA	NA
С	In clearing across small creek south of tailings dam, upstream of watergate	10	Steel	74.27 (pump top?)	26.07
D	In pumphouse above (east of) tailings pond dam, potable supply well, installed in February 1986	10	Steel	341.56	247.54
E	"MW-1" just off road on broad top level, ESE of pumphouse	2	PVC	255.31	80.28
F	2-inch PVC well on edge of slope above (north of) Carney Cr.	2	PVC	216.29	215.9
G	Near the headwaters of Carney Creek	Unknown	Unknown	Unknown	Unknown
Н	West of mine	2	PVC	Unknown	Unknown
l	Northeast of mine within upper Fleetwood Creek drainage	Unknown	Unknown	Unknown	Unknown
J	North of mine on hillside	Unknown	Unknown	Unknown	Unknown

TD = total depth
BTOC = below top of casing
SWL = surface water level

TABLE 6-1 **Analyses of Groundwater for Non-Asbestos Parameters** 

Parameter	EPA Analytical Method Reference Number	Target MDL (1) (ug/L)	Required PQL (2) (ug/L)	Groundwater Quality Standard (3) (ug/L)	Human Health RBCs (4) (ug/L)
D 45 A- 10/-4		easurements			
Depth to Water	Water Level Indicator			<del></del>	<del></del>
Temperature	YSI Model 5563 field meter or		+/-1°C	0.51+0.5	<del>-</del>
pH			+/- 0.1 pH unit	6.5 to 8.5	
Specific conductance	equivalent YSI 55 probe or equivalent		10 µmhos		
Dissolved oxygen		<del></del>	1 mg/L		
Turbidity	HF Scientific Model DRT-15C field probe or equivalent	_	5 NTU		
		st Metals/Metalloids			
Aluminum	6010B/6020/200.7/200.8	0.5	87	1	3650
Antimony	6010B/6020/200.7/200.8	0.05	6	6	1.46
Arsenic	6010B/6020/200.7/200.8	0.1	0.45	50	0.45(c)
Barium	6010B/6020/200.7/200.8	0.5	730	2000	730
Beryllium	6010B/6020/200,7/200.8	0.02	4	4	7.3
Boron	6010B/6020/200.7/200.8	5	730	1	730
Cadmium	6010B/6020/200.7/200.8	0.1	1	5	1.83
Calcium, dissolved	6010B/6020/200.7/200.8	100	1,000		
Chromium	6010B/6020/200.7/200.8	1	10	100	10.95
Cobalt	6010B/6020/200.7/200.8	0.2	10		
Copper	6010B/6020/200.7/200.8	0.6	146	1300	146
Iron	6010B/6020/200.7/200.8	5	300	300	2555
Lead	6010B/6020/200.7/200.8	0.2	15	15	
Magnesium, dissolved	6010B/6020/200.7/200.8	100	1,000		
Manganese	6010B/6020/200.7/200.8	0.05	50	50	73
Mercury	6010B/6020/200.7/200.8	0,03	2	2	
Nickel	6010B/6020/200.7/200.8	1	73	100	73
Potassium, dissolved	6010B/6020/200.7/200.8	100	1,000		
Selenium	6010B/6020/200.7/200.8	0.02	18	50	18.25
Silver	6010B/6020/200.7/200.8	0.05	18	100	18.25
Sodium, dissolved	6010B/6020/200.7/200.8	100	1,000		
Thallium	6010B/6020/200,7/200.8	0.1	0.255	2	0.255
Vanadium	6010B/6020/200.7/200.8	1	3.65	<del>_</del>	3.65
Zinc	6010B/6020/200.7/200.8	1	1,095	5000	1095
		chemistry	1 48 - 89 1	4669	
Gross Alpha	900.0	1 pCi/L	15 pCi/L	15 pCi/L	
Gross Beta	900.0	1 mrem	4 mrem	4 mrem	
Moletile Detector to the section of		Compounds			
Volatile Petroleum Hydrocarbons	SW8015M (6)MADEP-VPH-04-1.1 (7)	70	300		
Extractable Petroleum Hydrocarbons	SW8015M (6)MADEP-VPH-04-1.1 (7) Ma	70 Jor lons	300		
Fluoride	300.0	16	219	4000	219
Chloride	300.0	5	10		
Nitrate, as N	353.2	9,5	5840	10000	5840
Nitrite, as N	353.2	7.5	365	1000	365
Alkalinity (carbonate and bicarbonate)	SM 2320B	2000	4000		
Sulfate	300.0	70	500000	500000	
		Other			
Cyanide, Total	Kelada mod.	11	200	200	
Total Dissolved Solids (TDS)	SM 2540C/D	1000	500000	500000	
Total Suspended Solids (TSS)	SM 2540D	0	500000	500000	

- Notes: 1. MDL = method detection limit,
- 2. PQL = practical quantitation limit. Required PQLs are maximum quantitation limits based on comparison to groundwater quality standards and human-health riskbased concentrations for tap water, if available. PQLs given for parameters without regulatory or risk-based criteria are from Energy Labs, Inc., Helena, MT.
- 3. MCLs from Montana DEQ 1999, Public Water Supply Section, Community Summary: Drinking Water Regulations for Community Public Water Supplies, September, 1999, and current EPA Drinking Water Standards (primary and secondary)
- 4. EPA Region 3 Tap Water Human Health RBCs at HQ = 0.1 for non-cancer risk and 10-5 risk level for cancer. Cancer RBCs are indicated with (c).
- 5. Analyze for total metals concentrations, except where noted.
- 6. SW8015M will be used as a screening method to determine if more detailed analysis by MA-DEP EPH is needed.
- 7. Massachusetts Department of Environmental Quality Methods (http://www.mass.gov/dep/cleanup/laws/vph\_eph.htm#ana).

Table 7-1. Summary of Field Quality Control Samples

Medium	Field QC Sample Type	Minimum Collection Frequency	Analyses to be Performed	Acceptance Criteria	Corrective Action
Ambient air	Lot Blank	2 per cassette lot	TEM	No asbestos structures detected	Discard entire lot
	Field Blank	1 per sampling round (1 per 2 weeks)	TEM	No LA structures detected	Assign qualifier to analyte(s) in field samples associated with field blank (same day, same team)
	Field Duplicate	1 per 10 field samples (10%)	ТЕМ	None	None
Groundwater			ТЕМ	No LA structures detected	Assign qualifier to analyte(s) in field
	Field Blank 1 per 10 field samples (10%)	Metals, Anions, VPH, EPH, DOC (SW only)	Non-detect ( <mdl) for all target analytes</mdl) 	samples associated with field blank (same day, same team)	
	Trip Blank	1 per cooler of samples for VOC and VPH analyses	SW 8260 or MA-DEP-VPH (a)	Non-detect ( <mdl) for all target analytes</mdl) 	Assign qualifier to analyte(s) in field samples associated with trip blank (same cooler)
		1 per sampling team	TEM	No LA structures detected	Assign qualifier to analyte(s) in field samples associated with field blank (same day, same team)
	Equipment Rinsate Blank	Equipment per day (if non-	Metals, Anions, VPH, EPH, DOC (SW only)	Non-detect ( <mdl) for all target analytes</mdl) 	
		1 per 10 field	TEM	<5% statistically different	Assign qualifier to
	Field Split	samples (10%)	Same analyte list as original sample	20% RPD for target analytes	analyte(s) in parent field sample
	Performance Evaluation (PE)	4 PE samples	Inorganic and organic analytes	(b)	Assign qualifier to field samples for analyte(s) outside of acceptance criteria

<sup>(</sup>a) depending on analyses requested with associated samples
(b) meet analyte-specific criteria specified by QATS certification program

Table 7-2. Summary of Laboratory Quality Control Measures by Analysis

Analytical Method <sup>(n)</sup>	OC Element	Frequency	Acceptance Criteria	Corrective Action
ICP Metals SW-846 6010B (and EPA 200.7 for aqueous samples)	Initial calibration (1 point + blank minimum)	Daily prior to analysis	Correlation coefficient (r) ≥0.995	Recalibrate
	Interference check standard (ICS)	analytical run	Results +/- 20% of true value	Terminate analysis     Recalibrate instrument     Reanalyze all samples back to last acceptable ICS
		analysis	Results <10% from calibration standard	Reanalyze ICV     Recalibrate, if ICV still out
	(CCV)	Every 10 samples and end of analytical sequence	Results < 10% from calibration standard	Reanalyze affected samples back to the last acceptable CCV
	Calibration blank - Initial calibration blank (ICB), Continuing calibration blank (CCB)	After initial calibration verification, each subsequent calibration verification, and at the end of the run	<3x the Method detection limit (MDL)	Reanalyze blank     Clean system     Reanalyze all samples back to last acceptable blank
	Method blank	I per preparation batch (≤20 samples)	< Reporting limit	Reanalyze method blank. If fails, analyze a calibration blank Reprep/reanalyze analytical batch as appropriate
	Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery +/-25% of actual value	Assess data (4 x rule)     If LCS recoveries are within acceptance criteria, then matrix interference may be suspected     Reanalyze reprep once if matrix is not a factor     Narrate all outliers
	Matrix spike duplicate (MSD)	l per preparation batch (≤20 samples)	RPD <20%	Same as MS
	Laboratory Control Sample (LCS)	I per preparation batch (≤20 samples)	% Recovery +/- 20% of actual value	Reanalyze LCS Reprep/reanalyze LCS and affected samples Narrate all outliers
ICP-MS Metals SW-846 6020 (and EPA 200.8 for aqueous samples)	Mass calibration and resolution check ( 4 replicates )	Daily prior to analysis	Mass calibration < 0.1 amu; resolution <0.9 amu at 10% peak height; RSD <5%	Recalibrate
	Initial multipoint calibration (I point + blank minimum); average of 3 integrations	Daily prior to analysis	None	• None
	Initial calibration verification (ICV); mid-level standard second source	After calibration, prior to sample analysis	± 10% from true value	Reanalyze ICV     Recalibrate, if ICV still out
	Continuing calibration verification (CCV)	Every 10 samples and end of run sequence	± 10% from true value	Reanalyze affected samples back to the last acceptable CCV

Analytical Method <sup>(a)</sup>	QC Element	Frequency	Acceptance Criteria	Corrective Action
ICP-MS Metals SW-846 6020 (and EPA 200.8 for aqueous samples)	Interference check solution	At beginning of analytical sequence or once every 12 hours, whichever is more frequent	Recoveries +/- 20% of theoretical value	Internal QC review only; flag data to indicate interference
	Internal Standards	Every CCV, ICB/CCB	Recoveries +/- 20% of initial calibration	Recalibrate and verify calibration     Reanalyze affected samples
		Every sample	Recoveries 30-120% for samples	Dilute sample 5x and reanalyze     Repeat until within limits
	Calibration blank Initial calibration blank (ICB) Continuing calibration blank (CCB)	After initial calibration and each subsequent calibration verification	< 3 x Method detection limit (MDL)	Reanalyze blank     Clean system if still out     Reanalyze affected samples back     to the last acceptable CCB
	Method blank	l per preparation batch (≤ 20 samples)	< Reporting limit	Reanalyze method blank.     If fails, analyze a calibration blank     Reprep/reanalyze analytical batch as appropriate
	Matrix spike (MS)	1 per preparation batch (≤ 20 samples)	% Recovery +/- 25% of true value	Assess data     Reanalyze MS if matrix is not a factor
	Matrix spike duplicate (MSD) or Matrix duplicate (MD)	1 per preparation batch (≤ 20 samples)	RPD $< 20\%$ (for values $> 100 \times MDL$ )	Same as MS
	Post-digestion spike addition	As necessary to assess matrix interference	% Recovery +/- 25% of actual value	Perform dilution test     Or, perform method of standard addition
	Dilution test	1 per 20 samples	% Recovery +/- 10% of true value	Use method of standards addition
	Laboratory control sample (LCS)	I per preparation batch (≤ 0 samples)	%Recovery within +/- 20% of true value	Reanalyze LCS     Reprep/reanalyze LCS and affected samples     Narrate all outliers
Mercury SW-846 7470A/7471A	Initial multipoint calibration (3 point + blank minimum)	Daily, prior to analysis	Correlation coefficient (r) ≤0.995	Recalibrate
	Initial calibration verification (ICV); mid-level standard	After calibration, prior to sample analysis		Reanalyze ICV     Rerun initial calibration
	Continuing calibration verification (CCV); mid-level standard	Every 10 samples and at end of analytical sequence	± 20% of true value	Reanalyze affected samples back to last acceptable CCV
	Calibration blank (ICB/CCB)	After calibration, and after each subsequent calibration verification	< Reporting limit	Reanalyze blank     Clean system if still out     Reanalyze affected samples back     to last acceptable CCB
	Method blank	I per preparation batch (≤20 samples)	< Reporting limit	Reanalyze method blank.     If fails, analyze a calibration blank     Reprep/reanalyze analytical batch as appropriate

Analytical Method <sup>(a)</sup>	QC Element	Frequency	Acceptance Criteria	Corrective Action
Mercury SW-846 7470A/7471A	Matrix spike (MS)	I per preparation batch (≤20 samples)	% Recovery +/- 25% of true value	If LCS recoveries are within acceptance criteria, matrix interference may be suspected     Reprep/reanalyze once if problem cannot be attributed to matrix     Narrate all outliers
	Matrix spike duplicate (MSD)	l per preparation batch (≤20 samples)	RPD < 20%	Same as MS
	Laboratory control samples (LCS)	1 per preparation batch (≤20 samples)	%Recovery within +/- 20% of true value	Reanalyze LCS     Reprep/reanalyze LCS and affected samples     Narrate all outliers
SW-846, 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)	Tune instrument with a 4-bromofluorobenzene standard (BFB)	Every 12 hours	Must meet key ions and ion abundance criteria established by method.	
	Initial multi-point calibration; 5 point minimum. Lowest point at or below PQL. Includes calibration check compounds (CCC) and system performance check compounds (SPCC), and Internal Standards Compounds (IS).	Prior to analysis, and as required	RSD< 30 % for CCC; Average RF ≥ 0.1 for SPCC (≥0.3 for chlorobenzene, 1,1,2,2-Tetrachloroethane) If % RSD < 15% average RF may be used; linear calibration required	Evaluate system     Repeat calibration
	Continuing calibration verification (CCV): CCC, SPCC, and IS	Every 12 hours	Percent difference <20% for CCC; RF ≥0.1 for SPCC (≥0.3 for chlorobenzene and 1,1,2,2-Tetrachloroethane).	Evaluate system/standard     Reanalyze calibration check standard     Repeat initial calibration
	IS	Every sample, method blank, LCS, MS/MSD	Retention time for each internal standard must be within 30 seconds of most recent CCV and the EICP area for all internal standards must be within - 50% to +100% of the most recent CCV.	Evaluate system     Reanalyze sample once     Re-extract/reanalyze sample once     If due to media interference report both sets of data     Narrate all outliers
	Method blank	1 per preparation batch (≤20 samples)	< Reporting limit	Reanalyze method blank     Reanalyze batch
	Internal standards	Every sample, method blank, LCS, and MS/MSD	Retention time for each internal standard must be within 30 seconds of most recent CCV and the EICP area for all internal standards must be within - 50% to +100% of the most recent CCV	Evaluate system/standard     Reanalyze samples     If still out, report both sets of data     Narrate all outliers

Analytical Method <sup>(a)</sup>	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)	Surrogate spike	Every sample, method blank, LCS, MS/MSD	No more than one surrogate outside QC acceptance criteria. No surrogate below 10% recovery.	Reanalyze sample once Re-extract and reanalyze if > 1 surrogate outside QC acceptance limits If still out, report both sets of data Narrate all outliers
		1 per preparation batch (≤20 samples)	Percent recovery within QC acceptance criteria (Attachment X)	Assess data (4x rule)     If LCS and surrogate recoveries are within acceptance criteria matrix interferences may be suspected     Reprep/reanalyze once if matrix is not a factor     Narrate all outliers
ļ	Matrix spike duplicate (MSD) or	1 per preparation batch	% Recovery and/or RPD within QC	Same as MS
	Matrix Duplicate (MD)	(≤20 samples)	acceptance criteria (Attachment X)	
		1 per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria (Attachment X)	Reanalyze LCS     Reprep/reanalyze LCS and all associated samples     Narrate all outliers
SW-846 8270C Semi-Volatiles by GC/MS	decafluorotriphenylphosine (DFTPP) standard	Every 12 hours	Must meet the ion abundance criteria specified in the Degradation of DDT ≤ 20% Benzidine and PCP present at normal response without excessive tailing	Retune instrument     Repeat standard analysis     Perform injection port, column maintenance as necessary
	Initial calibration (5 point minimum); includes Calibration Check Compounds (CCC), System Performance Calibration Check (SPCC), and Internal Standard Compounds (IS)	Prior to analysis and as required	% RSD for CCC ≤30%; average RF ≥0.05 for SPCC If % RSD ≤15 % average RF may be used; linear calibration required	Evaluate the system     Repeat calibration
	Continuing calibration verification (CCV); includes CCC, SPCC, and IS	Every 12 hours	CCV percent difference for CCC ≤30%; RF ≥0.05 for SPCC EICP area of each internal standard - 50% to +100% of all IS areas in most recent CCV. Retention time for each internal standard must be within 30 seconds of most recent CCV	Evaluate system/standard     Reanalyze calibration check standard     Repeat the initial calibration as necessary
	Method blank	I per preparation batch (≤20 samples)	<reporting limit<="" td=""><td>Reanalyze blank     Reprep/reanalyze blank and all associated samples</td></reporting>	Reanalyze blank     Reprep/reanalyze blank and all associated samples

Analytical Method <sup>(a)</sup>	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846 8270C Semi-Volatiles by GC/MS		Every sample, method blank, LCS and MS/MSD	The EICP area for all internal standards must be within -50% and +100% of most recent CCV Retention time for each internal standard must be within 30 seconds of most recent CCV	Evaluate system/standard     Reanalyze the sample     If still out, report both sets of data
		Every sample, method blank, LCS and MS/MSD	No more than one surrogate per fraction outside of acceptance criteria (Refer to Table B1-a) No surrogate below 10% recovery	Reanalyze sample once     Re-extract and reanalyze if >1     surrogate per fraction outside acceptance limits     Narrate all outliers
•	Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria (Attachment X)	<ul> <li>Assess data (4x rule)</li> <li>Reanalyze once; if matrix is not a factor</li> <li>If LCS and surrogate recoveries are within acceptance criteria matrix interference maybe suspected</li> <li>Narrate all outliers</li> </ul>
	Matrix spike duplicate (MSD) or Matrix Duplicate (MD)	1 per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria (Attachment X)	Same as MS
	Laboratory control sample	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria for all spiked analytes (Attachment X)	Reanalyze LCS     Re-prep/reanalyze LCS and all associated samples     Narrate all outliers
SW-846 8082 Polychlorinated biphenyls (PCBs) by Gas Chromatography	Lowest standard at or below PQL; Expected Aroclors or Aroclor 1016/1260 five-point if unknown with single-point mid-level standards for other Aroclors for pattern recognition and retention times, or		RSD <20%, average calibration factor or response factor(a) may be used; linear calibration required	Evaluate the system     Repeat initial calibration
	Initial calibration verification (ICV) Mid level standard Expected Aroclors or Aroclor 1016/1260 if unknown	Prior to each 12 hour shift	% Difference ≤15% of expected concentration compared to response from ICAL	<ul><li>Evaluate system/standard</li><li>Reanalyze ICV standard</li><li>Repeat initial calibration</li></ul>
	Continuing calibration verification (CCV) Mid level standard Expected Aroclors or Aroclor 1016/1260 if unknown	After every 20 samples and at the end of the analytical sequence	% Difference ≤15% of expected concentration compared to response from ICAL for each bracketing standard	Evaluate system/standard     Reanalyze CCV and samples back to last acceptable CCV
	Retention time windows	Established with each new column installation Updated with each daily initial calibration standard	Retention times must be within retention time window established by the daily initial calibration standard Every CCV and every sample	Evaluate system/standard; pattern recognition may be sufficient     Reanalyze CCV/affected samples

Analytical Method <sup>(a)</sup>	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846 8082 Polychlorinated biphenyls (PCBs) by Gas Chromatography	Method Blank	1 per preparation batch (≤20 samples)	<reporting limit<="" td=""><td>Reanalyze blank     Re-prep/reanalyze blank and     associated samples</td></reporting>	Reanalyze blank     Re-prep/reanalyze blank and     associated samples
	Surrogate spike DCB (for Aroclors) TCMX (for PCB congeners)	Every sample, method blank, LCS and MS/MSD	% Recovery within QC acceptance criteria (Attachment X)	Re-extract/reanalyze once     If still out, report both sets of data     Narrate all outliers
	Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery within QCacceptance criteria (Attachment X)	Assess data (4x rule)     If LCS and surrogate recoveries are within acceptance criteria matrix interference maybe suspected     Re-extract/reanalyze if matrix is not a factor     Narrate all outliers
	Matrix spike duplicate(MSD) or Matrix duplicate (MD)	I per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria (Attachment X)	Same as MS
	Laboratory control sample(LCS)	I per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria (Attachment X)	Reanalyze LCS Re-prep/reanalyze LCS and all associated samples Narrate all outliers
SW-846 8081A Organochlorine Pesticides by Gas Chromatography	Column Evaluation Mix	Prior to analysis, both initial and daily	Degradation of DDT and Endrin < 15%	Evaluate the system     Repeat standard
	Initial calibration (5 point minimum) Lowest at or below PQL Mid level multi-component standards for pattern recognition and retention times	Prior to analysis and as required	RSD < 20%, average CF may be used; linear calibration required	Average RSD <20% across all analytes may be used if any analyte fails     Evaluate the system     Repeat initial calibration
	Initial calibration verification (ICV) Mid level standard Expected multi-component compounds	Prior to each 12 hour shift	% Difference ≤15% of expected concentration compared to response from ICAL	Average % difference ≤15% across all analytes may be used if any analyte fails     Evaluate system/standard     Reanalyze ICV standard     Repeat initial calibration
	Continuing calibration verification (CCV) Mid level standard Expected multi-component compounds	After every 20 samples and at the end of the analytical sequence	% Difference ≤15% of expected concentration compared to response from ICAL for each bracketing standard	<ul> <li>Average % difference ≤15% across all analytes may be used if any analyte fails</li> <li>Evaluate system/standard</li> <li>Reanalyze CCV and affected samples</li> <li>For CCV with response &gt; initial calibration response and % difference &gt;15%, samples need not be reanalyzed if no target compounds are detected</li> </ul>

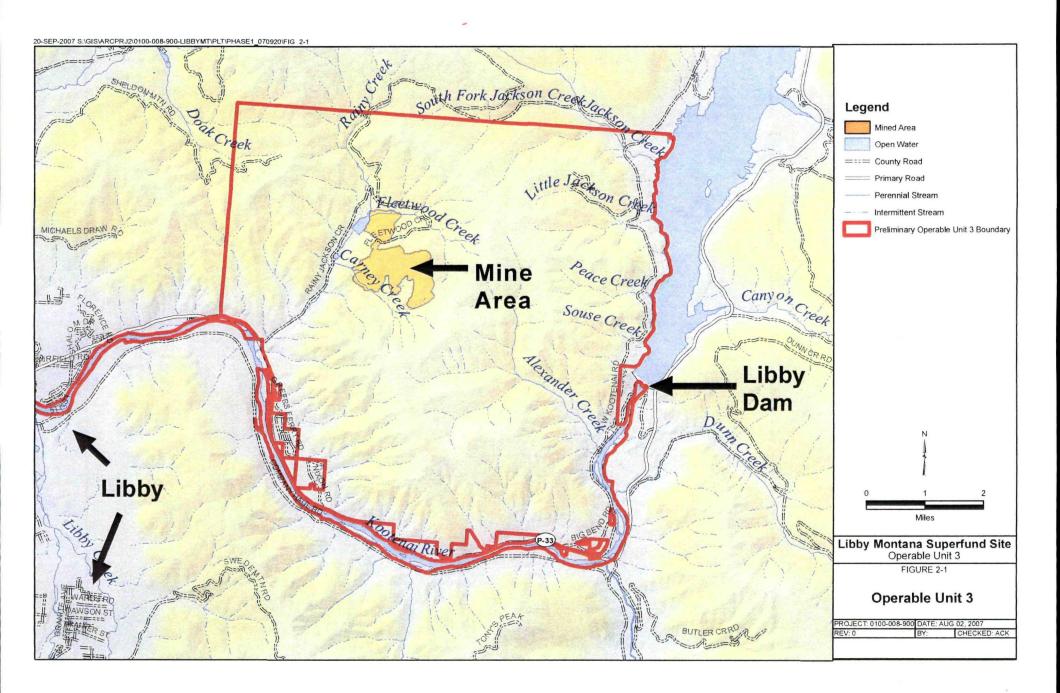
Analytical Method <sup>(a)</sup>	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846 8081 Organochlorine Pesticides by Gas Chromatography	Retention time windows	Established with each new column installation Updated with each daily initial calibration standard	Retention times must be within retention time window established by the daily initial calibration standard Every CCV and every sample	Evaluate system/standard; pattern recognition may be sufficient for multi-component compounds only     Reanalyze CCV/affected samples
	Method Blank	1 per preparation batch (≤ 20 samples)	<reporting limit<="" td=""><td>Reanalyze blank     Re-prep/reanalyze blank and     associated samples</td></reporting>	Reanalyze blank     Re-prep/reanalyze blank and     associated samples
	Surrogate spike DCB and TCMX	Every sample, method blank, LCS and MS/MSD	% Recovery within QC acceptance criteria (Attachment X). One surrogate must fall within established control limits	Re-extract/reanalyze once     If still out, report both sets of data     Narrate all outliers
	Matrix spike (MS)	I per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria (Attachment X)	Assess data (4 x rule)     If LCS and surrogate recoveries are within acceptance criteria, matrix interference maybe suspected     Re-extract/reanalyze once if matrix is not a factor     Narrate all outliers
	Matrix spike duplicate(MSD) or Matrix Duplicate (MD)	1 per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria (Attachment X)	Same as MS
	Laboratory control sample (LCS)	1 per preparation batch (≤ 20 samples)	% Recovery within QC acceptance criteria (Attachment X)	Reanalyze LCS     Re-prep/reanalyze LCS and all associated samples     Narrate all outliers
SW-846 8141A Organphosphorus Pesticides by Gas Chromatography	Initial calibration (5 point minimum) Lowest at or below reporting limit (RL)	Prior to analysis and as required	If %RSD < 20% average RF may be used If linear regression used r > 0.995 or R2 > 0.990 Alternate evaluation: Mean % RSD for all target analytes < 20% with no individual compound > 40%	Average RSD <20% across all analytes may be used if any analyte fails     Evaluate the system     Repeat initial calibration
	Initial calibration verification (ICV), second source Mid level standard	Prior to every analytical sequence	% Difference ≤15% of expected concentration compared to response from ICAL	Evaluate system/standard     Reanalyze ICV standard     Repeat initial calibration
	Continuing verification standard (CVS) Mid level standard	After every 10 samples and at the end of the analytical sequence	%D or % Drift >15%	Evaluate system/standard     Repeat sample analysis to last acceptable CVS
	Retention time windows	Established with each new column installation Updated with each daily initial calibration standard	Retention times must be within retention time window established by the daily initial calibration standard Every CVS and every sample	Evaluate system/standard; pattern recognition may be sufficient for multi-component compounds only     Reanalyze CVS/affected samples
	Target analyte confirmation	All detected analytes	RPD < 40%	If greater than 40% qualify data as estimated

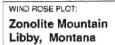
Analytical Method <sup>(a)</sup>	QC Element	Frequency	Acceptance Criteria	Corrective Action
Pesticides by Gas	Method Blank	1 per preparation batch (≤ 20 samples)	< ½ RL	Reanalyze blank     Reprep/reanalyze blank and     associated samples
	Surrogate spike	Every sample, method blank, LCS and MS/MSD	% Recovery within QC acceptance criteria (Attachment X)	<ul> <li>Reanalyze</li> <li>Reprep/reanalyze once</li> <li>If still out, report both sets of data</li> <li>Narrate all outliers</li> </ul>
	Matrix spike (MS)	I per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria (Attachment X)	<ul> <li>Reanalyze</li> <li>Reprep/reanalyze once</li> <li>If still out, report both sets of data</li> <li>Narrate all outliers</li> </ul>
	Matrix spike duplicate(MSD)	1 per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria (Attachment X)	Same as MS
	Laboratory control sample (LCS)	I per preparation batch (≤ 20 samples)	% Recovery within QC acceptance criteria (Attachment X)	<ul> <li>Reanalyze LCS</li> <li>Reprep/reanalyze LCS and all associated samples</li> <li>Narrate all outliers</li> </ul>
SW-846 8151A Organochlorine Herbicides and Pentachlorophenol by Gas Chromatography	Initial calibration (5 point minimum) Lowest point at or below PQL	Prior to analysis and as required	CF may be used; linear calibration required	<ul> <li>Average RSD &lt;20% across all analytes may be used if any analytes fail</li> <li>Evaluate the system</li> <li>Repeat initial calibration</li> </ul>
	Initial calibration verification (ICV) second source Mid level standard	Prior to each daily analytical sequence	% Difference ≤15% of expected concentration compared to response from ICAL	<ul> <li>Average %D ≤15% across all analytes may be used if any analytes fail</li> <li>Evaluate system/standard</li> <li>Reanalyze ICV standard</li> <li>Repeat initial calibration</li> </ul>
	Continuing calibration verification (CCV) Mid level standard	After every 20 samples and at the end of the analytical sequence	% Difference ≤15% of expected concentration compared to response from ICAL for each bracketing standard	Evaluate system/standard     Reanalyze CCV and all samples back to last acceptable CCV
	Retention time windows	Established with each new column installation Updated with each daily initial calibration standard	Retention times must be within retention time window established by the daily initial calibration standard Every CCV and every sample	Evaluate system/standard;     Reanalyze CCV and affected samples
	Method blank	1 per preparation batch (≤20 samples)	<reporting limit<="" td=""><td>Reanalyze blank     Re-prep/reanalyze blank and all associated samples</td></reporting>	Reanalyze blank     Re-prep/reanalyze blank and all associated samples
	Surrogate spike DCAA	Every sample, method blank, LCS and MS/MSD	% Recovery within project QC acceptance criteria (Attachment X)	Re-extract/reanalyze once     If still out, report both sets of data     Narrate all outliers

.

Analytical Method <sup>(a)</sup>	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846 8151A Organochlorine Herbicides and Pentachlorophenol by Gas Chromatography	Matrix spike (MS)	I per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria (Attachment X)	Assess data (4x rule)     If LCS and surrogate recoveries are within acceptance criteria, matrix interference maybe suspected     Re-exact/reanalyze once if matrix is not a factor     Narrate all outliers
	Matrix spike duplicate (MSD) or Matrix duplicate (MD)	1 per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria (Attachment X)	Same as MS
	Laboratory control sample (LCS)	1 per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria (Attachment X)	Reanalyze LCS     Re-prep/reanalyze LCS and all associated samples     Narrate all outliers
Total Cyanide SW-846 9012B	Initial calibration curve (six standards and a calibration blank)	Initial daily calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	<ul> <li>Correct problem then repeat initial calibration</li> </ul>
	Distilled standards (one high and one low)	Once per initial calibration	Cyanide within ±10% of true value	Correct problem then repeat distilled standards
	Second-source calibration verification	One per preparation batch (<20 samples)	Cyanide within ±15% of expected value	Correct problem then repeat initial calibration
	Method blank	One per analytical batch	No analytes detected ≥ Reporting Limit	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes	One per preparation batch (<20 samples)	QC acceptance criteria (Attachement X)	Correct problem then reanalyze     If still out, reprep and reanalyze     the LCS and all samples in the     affected AFCEE batch
	MS/MSD	One per preparation batch (<20 samples)	QC acceptance criteria (Attachement X)	• None
Gross Alpha and Gross Beta SW-846-9310	Initial calibration with standard reference materials	Daily before sample analysis	Analytical method control limits	Correct problem and repeat calibration
	Method Blank	One per analytical batch	No analytes detected ≥ Reporting Limit	Identify and reduce contamination then reanalyze
	Analytical Duplicate	One per analytical batch	RPD < 20	Evaluated problem and correct the reanalyze
	Spiked Sample or standard reference material	One per analytical batch	80-120% recovery	Evaluated problem and correct the reanalyze

EICP Extracted ion current profile QC Quality control RF Response factor RSD Relative standard deviation





DISPLAY: Wind Speed Direction (blowing from)

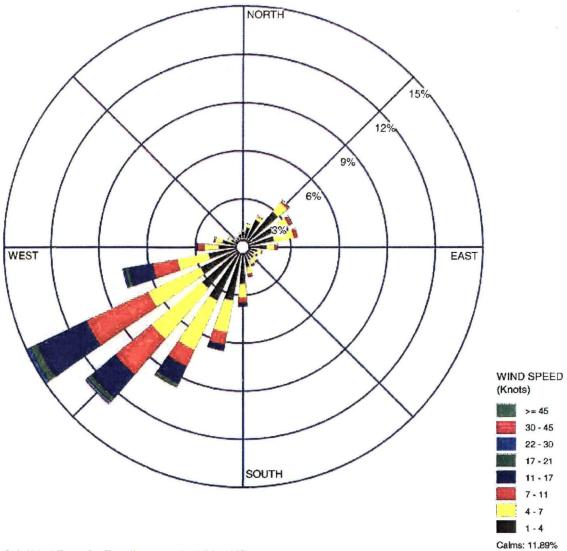
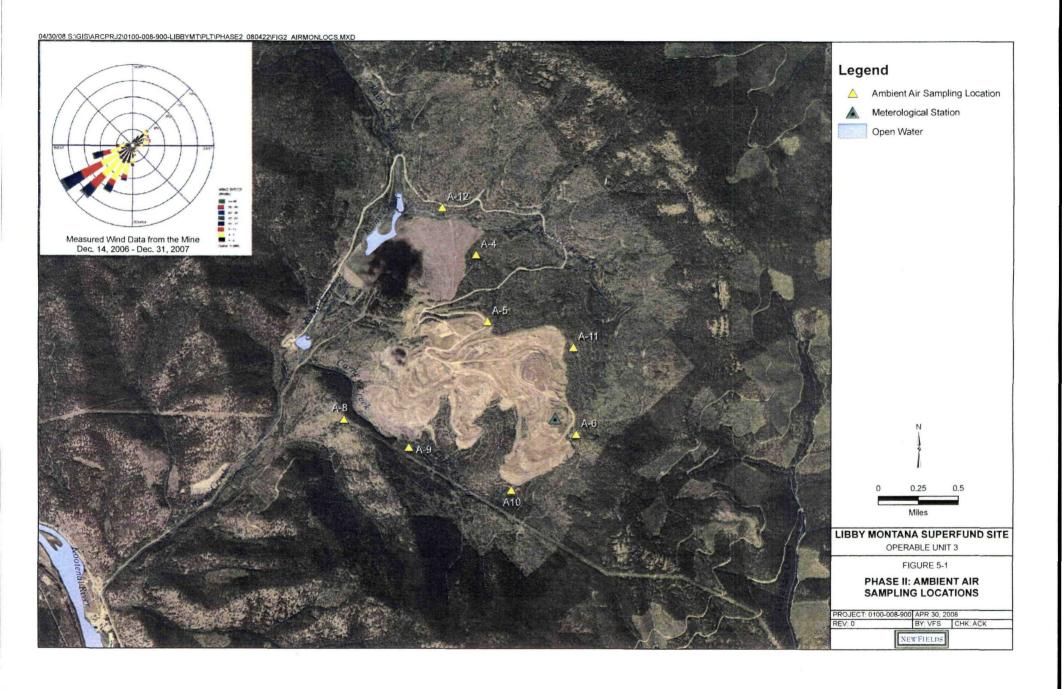
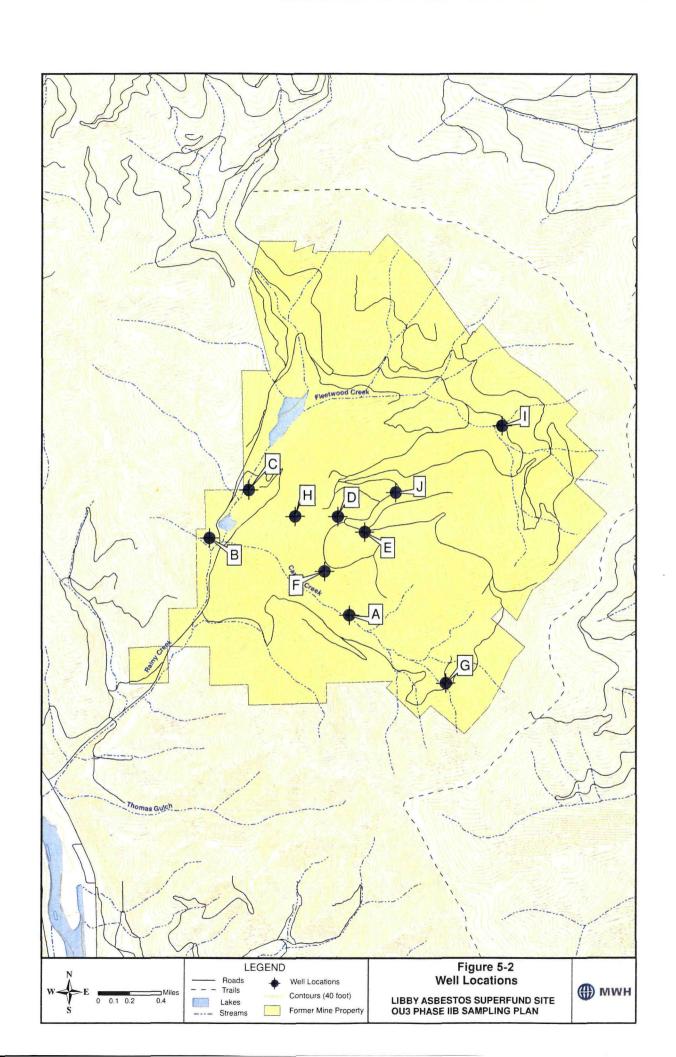


Figure 3-1 Wind Rose for Zonolite Moutain, Libby, MT

COMMENTS:	DATA PERIOD:	COMPANY NAME:						
Cumulative Data December 14, 2006 to	2006-2007 Jan 1 - Dec 31	W.R. Grace						
December 31, 2007	00:00 - 23:00	MODELER:						
Missing data from November 17 2007 to December 14 2007	CALM WINDS:	TOTAL COUNT:						
	AVG. WIND SPEED:	DATE: 1/18/2008	PROJECT NO.:					
4								





# ATTACHMENT A

## STANDARD OPERATING PROCEDURES

SOP Description	SOP ID					
Groundwater Sampling for Chemical Analysis	No. 6 (Rev. 0)					
Equipment Decontamination	No. 7 (Rev. 0)					
Sample Handling and Shipping	No. 8 (Rev. 0)					
Field Documentation	No. 9 (Rev. 4)					
Field Equipment Calibration	No. 10 (Rev. 1)					
GPS Data Collection	No. 11 (Rev. 1)					
Investigation Derived Waste (IDW) Management	No. 12 (Rev. 0)					
Groundwater Elevation Measurements	No. 13 (Rev. 0)					
Collection of Outdoor Ambient Air Samples	AMB-LIBBY-OU3 (Rev. 1)					
Well Redevelopment	Libby Well Redevelop (Rev. 0)					

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Date: September 26, 2007

OU3 SOP 6 (Rev. 0)

Title: GROUNDWATER SAMPLING

APPROVALS:

TEAM MEMBER

SIGNATURE/TITLE

DATE

**BPA Remedial Project Manager** 

**SOP Author** 

9/26/07

9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	

OU3 SOP 6 Rev. No. 0

Date: September 26, 2007 Page 1 of 14

1.0 INTRODUCTION

This standard operating procedure (SOP) is intended to serve as a reference for the proper

equipment and techniques for collecting groundwater samples for chemical analysis. The SOP is

based on MWH SOP-11, Groundwater Sampling, Revision 1.0, March 2004, modified for use at

the Libby Asbestos Superfund Site OU3. The purpose of this SOP is to enable the user to collect

representative and defensible groundwater samples, and to plan the field sampling effort. These

procedures are applicable when referenced by a monitoring or field sampling plan (FSP).

To be valid, a groundwater sample must represent the water-bearing unit being sampled. The

physical, chemical, and bacteriological integrity of the sample must be maintained from

collection to analysis in order to minimize changes in water quality. Acceptable equipment for

sample collection from completed wells includes bailers, electrical submersible pumps and

peristaltic pumps. In order to obtain a representative sample of the groundwater, the sampler

must accomplish the following:

avoid collecting stagnant (standing) water in the well;

• avoid physically or chemically altering the water due to improper sampling techniques,

sample handling, or transport; and;

• document that proper sampling procedures have been followed.

This SOP describes groundwater sample collection methods only. Sample handling and shipping

procedures are described in SOP-8. Field documentation procedures are presented in SOP-9.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in groundwater sampling must follow health and safety protocols

described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they

cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when

embedded in the lung tissue can cause health problems. Significant exposure to asbestos

increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and

other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Annular Space: The space between the casing or well screen and the wall of the drilled hole, or

between the drill pipe and casing, or between two separate strings of casing. The annular space

may also be referred to as the annulus.

Aquifer: A geologic formation, group of formations, or part of a formation that is capable of

yielding economic quantities of water to a well or spring.

Check Valve: Ball and spring valves on bailers and pumps that are used to allow water to flow

in one direction only.

Conductivity: A measure of how well water can conduct an electrical current. It is the

reciprocal of resistivity.

Datum: An arbitrary surface (or plane) used in the measurement of heads (i.e., National

Geodetic Vertical Datum [NGVD], commonly referred to as mean sea level [msl]).

Dissolved Oxygen (DO): A measure of the quantity of oxygen dissolved in groundwater. DO

data is collected in the field using direct measure probes. DO is used to assess the balance

between oxygen-consuming and oxygen-producing processes.

**Drawdown:** The lowering of the potentiometric or piezometric surface in a well and aquifer due

to the discharge of water from the well.

Filter-Pack: Sand or gravel that is generally uniform, clean, and well rounded that is placed in

the annulus of the well between the borehole wall and the well screen to prevent formation

material from entering the well screen, and to stabilize the adjacent formation.

OU3 SOP 6

Rev. No. 0

Date: September 26, 2007

Page 3 of 14

Groundwater Sample: A sample collected from an aquifer for chemical analysis to support

remedial investigation, feasibility studies, treatability studies, remediation design and

performance assessment, waste characterization, etc.

Head-Space: The empty volume in a sample container between the water level and the cap.

Monitoring Well: A well that is constructed by one of a variety of techniques for the purpose of

extracting groundwater for physical, chemical, or biological testing, or for measuring water

levels.

Oxidation-Reduction Potential (ORP): A measurement of the reducing or oxidation potential

of a given system or medium. ORP data are collected in the field using direct measure probes.

Positive potentials indicate the system is oxidizing, while negative potentials indicate the system

is reducing.

Packer: A transient or dedicated device placed in a well or borehole that isolates or seals a

portion of the well, well annulus, or borehole at a specific level.

**Peristaltic Pump:** A low-volume pump that operates by suction lift.

Piezometer: A small-diameter well used to measure groundwater elevation and/or to collect

periodic groundwater samples.

Static Water Level: The elevation of the top of a column of water in a monitoring well or

piezometer that is not influenced by pumping or conditions related to well installation,

hydrologic testing, or nearby groundwater extraction.

Turbidity: Cloudiness in water due to suspended and colloidal organic and inorganic material.

Units are commonly in Nephelometric Turbidity Units (NTUs).

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated

with them. This list is not intended to be comprehensive and often, additional personnel may be

involved. Project team member information will be included in project-specific plans (e.g., work

plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult

the appropriate documents to determine project-specific roles and responsibilities. In addition,

one person may serve in more than one role on any given project.

Project Manager: Selects site-specific groundwater sampling program with input from key

project staff and applicable oversight agencies.

Quality Control Manger: Overall management and responsibility for quality assurance and

quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods,

performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer: Implements the

sampling program, supervises other sampling personnel, and ensures compliance with SOPs and

OA/OC requirements. Prepares daily logs of field activities.

Field Sampling Technician (or other designated personnel): Assists the FTL and/or

geologist, hydrogeologist, or engineer in the implementation of tasks and is responsible for the

proper use, and maintenance of groundwater sampling equipment.

5.0 MONITORING WELL SAMPLE COLLECTION PROCEDURES

5.1 Background

The methods and procedures described in this SOP were developed from these sources:

• U.S. Environmental Protection Agency, Region 9. Standard Operating

Procedure for the Standard/Well-Volume Method for Collecting a Ground-Water Sample from Monitoring Wells for Site Characterization.

OU3 SOP 6 Rev. No. 0

Date: September 26, 2007

. Deptember 20, 2007

Page 5 of 14

- U.S. Environmental Protection Agency, Region 1, 1996. Low Stress (low flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells.
- U.S. Environmental Protection Agency, May 2002. Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers.
- U.S. Geological Survey, variously dated, National field manual for the collection of water-quality data; U.S. Geological Survey Techniques of Water-Resources Investigations, Book 9, Chapters A1-A9. Available online at http://pubs.water.usgs.gov/twri9A.

### 5.2 Sampling Equipment

There are several types of equipment available for well purging and sampling. Table 1 provides a summary of the groundwater sampling equipment and their appropriateness for specific parameters or group of parameters. Refer to the field sampling plan (FSP) for the sampling equipment specified. If there are no specifications in the FSP, consult Table 1 based on factors such as depth and diameter of the well, the recharge capacity of the well, and the analytical parameters of interest.

In addition, the following equipment is needed to collect groundwater samples:

- Field notebook and forms, indelible marker
- Global Positioning System (GPS) unit
- Digital Camera
- Water level measuring probes
- Detergent solution (0.1-0.3 % Alconox)
- Distilled water
- Latex gloves
- Ziploc bags
- Paper Towel
- Chain of custody and sample labels

OU3 SOP 6

Rev. No. 0

Date: September 26, 2007

Coolers

Sample bottles

Plastic sheeting

Filters

Photo ionization detector

5.3 Sampling Procedures

Wells and piezometers located upgradient of the site will be sampled first and areas

downgradient of site thought to contain contamination will be sampled last. Upon arrival at the

well or piezometer, prior to groundwater purging or sampling, sampling personnel will perform

and document the following procedures.

1. Don appropriate health and safety equipment

2. Setup clean plastic sheeting in area for processing samples

3. Inspect the well or piezometer

4. Measure for organic vapors

5. Measure depth to groundwater (to nearest hundredth of a foot) from marker denoting

measuring point (generally a notch cut into the casing on the north side)

6. Measure total depth of well (to nearest hundredth of a foot) from same marker

7. Calculate required purge volume as described below in section 5.3.2

8. Calibrate the water quality measuring instrument according to the manufacture's

specifications

9. Measure initial water quality for pH, specific conductivity, temperature, turbidity,

ORP, and DO

OU3 SOP 6

Rev. No. 0

Date: September 26, 2007

Page 7 of 14

Document all organic vapor measurement, depth to water, total well depth, and initial water quality information on the monitoring form. Refer to the FSP do determine whether low-flow purging and sampling or volume based purging and sampling is specified. Perform the well purging and sampling according to the procedures specified in the following subsections.

### 5.3.1 General Criteria for LOW-FLOW Sample Collection

For wells that are sampled for regulatory compliance, a low flow sample collection technique shall be employed to ensure that representative groundwater samples are collected from each well. Additionally, low flow sampling is to be the preferred method for groundwater sampling unless site specific conditions warrant a volume based approach (as discussed in Section 5.3.2). A variable rate submersible centrifugal or positive displacement type pump will be used for purging and sampling; however, if the water table is less than 20 ft a peristaltic pump may be employed as long as the constituents measured are not influenced by negative pressures.

To limit the disturbance to the groundwater and to reduce the large volumes of purged water, monitoring wells can be sampled using low-flow pumping rates. Studies have shown that groundwater in the screened interval of a standard monitoring well can be representative of groundwater in the formation, even though stagnant water lies above in the casing. This occurs when flow is generally horizontal and naturally purges the screened interval. However, the insertion of a sampling device, such as a bailer, can disrupt this equilibrium and cause mixing of the screened and cased interval waters. The mixture of stagnant and screened interval water can even be forced into the aquifer, resulting in chemical effects that may affect data quality. Purging with a bailer increases the turbidity, which can cause adverse impacts on the sample quality. Low-flow sampling technique is an accurate and less invasive method for groundwater sampling.

The objective of low-flow sampling is a more passive approach to sample extraction with the ideal condition being an equilibrium in the intake velocity with the natural groundwater flow velocity. Flow rates from 0.2 to 0.5 liter/minute (l/min) are typical for low-flow sampling. When performed correctly, water sampled using the low-flow technique is produced from the screened interval without disturbing stagnant water in the well casing. By using low flow rates to

OU3 SOP 6 Rev. No. 0 Date: September 26, 2007

Page 8 of 14

stabilize drawdown at less than the goal of 0.30 feet, only screened interval water will be sampled. Drawdown in low yielding formations may be greater than the 0.30 feet.

The following procedures will be used while performing low-flow purging and sampling:

- 1. Slowly lower pump, or tubing inlet for a peristaltic pump into the well until the intake is at the midpoint of the screened interval.
- 2. Secure the pump and tubing to avoid shifting during purging
- 3. Place water level indicator down to the top of the static water table and record the depth to groundwater.
- 4. Begin purging groundwater at its lowest speed and adjust to a rate between 0.2 to 0.5 liter/minute, making sure to minimize drawdown to less than 0.3 feet as a goal, (Rates up to approximately 0.9 1/min may be feasible only if the drawdown continues to be less than 0.3 feet)
- 5. Measure and record the discharge rate, drawdown, and water quality parameters at intervals of 30 seconds to five minutes during purging.
- 6. Purge until three consecutive water quality readings, collected at intervals of at least five minutes, are within:

pΗ  $\pm 0.1$  units

Conductivity ±3%

Temperature ±1 degree Celsius

Dissolved Oxygen ±10% difference

Eh-ORP ±10 millivolts

**Turbidity** ±10 % NTU (when turbidity is greater then

10 NTUs).

It may be difficult to meet all of aforementioned criteria due to the characteristics of the water and the fact these criteria are near the accuracy of the meters themselves. If a minimum of one tubing volume (including the volume of the water in the pump and the flow through cell)

> OU3 SOP 6 Rev. No. 0

Date: September 26, 2007 Page 9 of 14

has been met and the parameters have generally stabilized, groundwater samples will be collected.

- 7. Collect and preserve samples for Volatile Organic Compounds (VOC) first. Unpreserved sample containers should be rinsed with the sample water prior sample collection
- 8. Label sample containers with the sample location and sample analysis information
- 9. Pack samples containers to avoid leakage or breakage during shipment
- 10. Store and ship samples on ice at 4 degrees Celsius. For further details on shipping and handling refer to SOP-12

If the well produces water at an extremely slow rate and excessive drawdown is occurring (> 0.30 feet.) reduce the flow rate or turn the pump off for 15 minutes and allow for recovery. If after pumping is resumed at a slower flow rate (e.g., 0.2 l/min), excessive drawdown continues to occur continue purging until the water level in the well nearly reaches the intake level of the pump. Once the water level reaches this depth, stop pumping and allow the water level to recover to 90 percent of the original static level. Continue purging and monitoring until three consecutive readings meet the stabilization criteria.

If under special circumstances the well does not recover to 90 percent within a normal workday, the well may be allowed to recover overnight and be sampled the following morning.

#### 5.3.2 VOLUME BASED Sample Collection

In instances where low-flow sampling is not possible or necessary based on site-specific conditions samples from wells will be collected using a volume based approach as follows:

7. Calculate the volume of water within the well bore using the following formula (or equivalent): 7.4805 ((D²π)/4) dH = volume (in gallons), where D = casing diameter in feet. (NOTE: This equation is used for grouted wells with short screens. For wells with long screens and/or ungrouted wells, the D = borehole diameter in feet). dH = the distance from well bottom to static water level in feet; and π = 3.1416

OU3 SOP 6

Rev. No. 0

Date: September 26, 2007

Page 10 of 14

- 8. Lower pump or bailer intake to a depth just above the screened interval in a manner consistent with that specified for the low-flow sampling above. This is to avoid exposing the screened interval to atmospheric conditions at drawdown and obtain the most representative sample.
- 9. Measure and record the initial depth to water and water quality
- 10. Bail or pump the well until at least three casing volumes has been removed AND the water quality parameters have stabilized as described in section (5.3.1). The purge rate must not reach a point where the recharge water is entering the well in an agitated manner and the water level in the well during purging should not be allowed to drop below the pump intake.
- 11. No more than six well volumes should be removed to prevent the effects of over pumping. If the indicator parameters have not stabilized following six well volumes the field instruments will be recalibrated and checked for possible malfunction. If no problems are found, sampling can be conducted; however, the project manager will be notified and all information will be recorded in the field notebook and/or field purge record.
- 12. If the yield of the well is low such that it can be bailed or pumped dry, then the recharged groundwater in the well will be considered representative regardless of the number of casing volumes of groundwater removed, since all standing water in the well has been replaced by recharge from the water-yielding zone. If a well is purged dry, the well can be sampled upon 90% recovery or after two hours, whichever occurs first.

#### 5.4 Sample Filtration

Refer to the FSP. When required, a field-filtered water sample will be collected using a disposable, in-line 0.45 µm filter. The water sample will be pumped through the filter attached directly to the discharge tubing. A peristaltic pump and a section of Tygon (polyvinylchloride) tubing or other appropriate method may be used if the sample is collected via bailer. The filter cartridge will be rinsed with an aliquot of 500 ml of sample prior to collection of sample in to the containers or as per the filter manufacturer's recommendations. Both the filter and tubing will be disposed of between samples.

OU3 SOP 6

Rev. No. 0

Date: September 26, 2007

Page 11 of 14

5.4 Sample Preservation and Storage

Refer to the FSP. If required by the project or analytical method, water samples submitted for

chemical analysis will be stored at 4 °C in ice-cooled, insulated containers immediately after

collection. Preservation and storage methods depend on the chemical constituents to be analyzed

and should be performed according to requirements stated in the FSP.

6.0 PURGE WATER HANDLING AND DISPOSAL

Because of the potential for spreading environmental contamination, proper purge water disposal

is a necessary part of well monitoring. All purge water will be contained in 55-gallon drums or

fixed above-ground storage tank (e.g., Baker Tank) pending analytical results. The collected

purge water will be properly disposed of according to the analytical results and any local, state, or

federal guidelines.

7.0 QUALITY ASSURANCE AND QUALITY CONTROL

Duplicate, equipment rinsate, and matrix spike samples will be collected at the frequencies

documented in the FSP. All sampling data must be documented in the field logbooks and/or

field forms, including rationales deviations from this SOP. The Field Team Leader or designated

QA reviewer will check and verify that field documentation has been completed per this

procedure and other procedures referenced herein. All equipment must be operated according to

the manufacturer's specifications, including calibration and maintenance.

8.0 DECONTAMINATION

All equipment used in the groundwater sampling process shall be decontaminated prior to field

use and between sample locations. Decontamination procedures are presented in SOP-7.

Personnel shall don appropriate personal protective equipment as specified in the health and

safety plan. Any investigation-derived waste generated in the sampling process shall be managed

in accordance with the procedures outlined in SOP-12.

OU3 SOP 6

Rev. No. 0

Date: September 26, 2007

Page 12 of 14

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OU3 SOP 6 Rev. No. 0 Date: September 26, 2007 Page 13 of 14

TABLE 1 GENERALIZED GROUNDWATER SAMPLING DEVICE MATRIX

					[	GROUNDWATER PARAMETERS													
	_						INORGANIC ORGANIC										RADIOA	CTIVE	BIOLOGICAL
		Device	Approx Max Sample Depth	Min Weil Diameter	Sample Delivery Rate or Volume*	EC	ρH	Redox	Major lons	Trace metals	Nitrate Fluoride	Dissolved gasses	Non- volatile	Volatile	TOC	тох	Radium	Gross Alpha & beta	Coliform becteria
1		Open Baller	no limit	1/2 in.	varlable	•	0	0	•	•	•	0	•	0	0	0	•	0	•
	GRAB	Point-Source Bailer	no (kmit	1/2 ln.	variable	•	•	•	•	•	•	0	•	•	•	•	. •	•	•
S	Θ	Syringe Sempler	no (lmit	1 1/2 in.	0.01-0.2 gal	•	•	•	•	•	•		•	•	0	0	•	•	•
끐	ī	Gear-drive	200 ft.	2 ln.	0-0.5 gpm	•	•	•	•	•	•	•	•	•	•	•	•	•	•
DEV	IVE SIBL	Bladder pump	400 ft.	3/4 in.	0-2 gpm	•	•	•	•	•	•	•	•	•	•	•	•	•	•
SAMPLING DEVICES	POSITIVE DISPLACEMENT (SUBMERSIBLE)	Electric Submersible	160 ft.	2 in.	0-1.2 gpm	•	•	•	•	•	•	•	•	•	•	•	•	•	a
		Piston pump (gas-drive)	500 ft.	1 1/2 in.	0-0.5 gpm	•	۰	0	•	•	•	۰	•	•	0	•	•	•	0
PORTABLE	SUCTION	Peristattic	26 ft.	1/2 in.	0.01-0.3 gpm	•	•	a	•	•	•	o	•		•	o	•		•
	ACT A	Gas-fift	vartable	1 in.	variable		•	0	0	•	a	0	•	0	0	۰	0	٥	0
CONT	GAS	Gas-drive	160 ft.	1 in.	0.2 gpm	•	0	0	•	0	•	•	•	0	0	•	•	0	0
SA	N SITU MPLING VICES**	Pneumetic	no fimit	na limit	0.01-0.13 gpm	•	•	•	•	•	•	q	•	۰	•	•	•	•	•

- \* Sampling devices on this chart are divided into two categories: 1. Portable devices for sampling existing monitoring wells, and 2. In sin monitoring devices (often multilevel) that are permanently installed.
- Sample delivery rates and volumes are arcrage ranges based on typical field conditions. Actual delivery more are a function of well dismeter, size and expacity of sampling device, hydrogeologic conditions, and depth to sampling point.
- I delicates device is generally suitable for application (securing device is cleaned and operated properly and is constructed of suitable materials.

  o Indicates device may be unsaitable or is unrested for application.

Source: Modified from Pohlmann and Hess, 1988

Based on Literature Review

OU3 SOP 6 Rev. No. 0 Date: September 26, 2007 Page Hof 14

Date: September 26, 2007

OU3 SOP 7 (Rev. 0)

Title: EQUIPMENT DECONTAMINATION

APPROVALS:

TEAM MEMBER

EPA Remedial Project Manager

SOP Author

SIGNATURE/TITLE

DATE

9/24/07

9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	_

OU3 SOP 7 Rev. No. 0 Date: September 26, 2007 Page 1 of 8

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is based on MWH SOP-02, Equipment

Decontamination, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund

Site OU3. Decontamination of drilling, sampling, and monitoring equipment is a necessary and

critical aspect of environmental field investigations. Proper decontamination is a key element in

reducing the potential for cross-contamination between samples from different locations, as well

as ensuring that samples are representative of the sampled materials. Improper decontamination

may result in costly re-collection and re-analysis of samples. All equipment used in the sampling

process will be properly decontaminated prior to the collection of each sample and after

completion of sampling activities.

The procedures outlined in this SOP will be followed during decontamination of field equipment

used in the sampling process, including drilling, soil/water sample collection, and monitoring

activities. Any deviations from these procedures will be noted in the field notebooks and

approved by the appropriate oversight agency, if significant. Three major categories of field

equipment, along with applicable decontamination methods for each, are discussed below.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in equipment decontamination must follow health and safety protocols

described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they

cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when

embedded in the lung tissue can cause health problems. Significant exposure to asbestos

increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and

other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Bailer: A cylindrical tool designed to remove material from a well. A valve at the bottom of the

bailer retains the contents in the bailer.

OU3 SOP 7

Rev. No. 0

Date: September 26, 2007

Page 2 of 8

Bladder Pump: Groundwater sampling equipment consisting of a flexible bladder, usually

made of Teflon<sup>®</sup>, contained within a rigid cylindrical body (commonly made of stainless steel).

The lower end of the bladder is connected to the intake port through a check valve, while the

upper end is connected through a second check valve to a sampling line that leads to the ground

surface.

Brass Sleeve: Hollow, cylindrical sleeves made of brass and used as liners in split-spoon

samplers for collection of undisturbed samples.

Auger Flight: An individual auger section, usually 5 feet in length.

Continuous Core Barrel: 3-5 foot long steel barrels that can be joined together to allow

continuous cores to be collected during a single run.

Drill Pipe: Hollow metal pipe used for drilling, through which soil and groundwater sampling

devices can be advanced for sample collection.

Peristaltic Pump: A low-volume suction pump. The compression of a flexible tube by a rotor

results in the development of suction.

Source Water: A drilling quality water source identified to be used for steam cleaning. This

source should be sampled at the beginning of each field program to set baseline concentrations.

Distilled Water: Commercially available water that has been distilled. Each batch of distilled

water should be analyzed to set baseline concentrations.

Hand Auger: A sampling tool consisting of a metal tube with two sharpened spiral wings at the

tip.

Split-Spoon Sampler: A sampling tool consisting of a thick-walled steel tube with a removable

head and drive shoe. The steel tube splits open lengthwise when the head and drive shoe are

removed.

Scoop: A sampling hand tool consisting of a small shovel- or trowel-shaped blade.

Submersible Pump: Groundwater sampling pump that consists of a rotor contained within a

chamber and driven by an electric motor.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated

with them. This list is not intended to be comprehensive and often, additional personnel may be

involved. Project team member information will be included in project-specific plans (e.g., work

plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult

the appropriate documents to determine project-specific roles and responsibilities. In addition,

one person may serve in more than one role on any given project.

Project Manager: Responsible for project implementation and coordination, selects project-

specific drilling and sampling methods, and associated decontamination procedures with input

from other key project staff, and appropriate oversight agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and

quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods,

performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer: Implements the

field program and supervises other sampling personnel, and ensures that SOPs are properly

followed. Prepares daily logs of field activities.

Field Sampling Technician (or other designated personnel): Assists the FTL, geologist,

hydrogeologist, or engineer in the implementation of tasks and is responsible for the

decontamination of sampling equipment.

5.0 DECONTAMINATION PROCEDURES

Drilling and sampling procedures require that decontaminated tools be employed in order to

prevent cross-contamination. The decontamination procedures described below will be followed

to ensure that only uncontaminated materials will be introduced to the subsurface during drilling

and sampling. The equipment decontamination process will be undertaken before and after each

use of the equipment and include either steam cleaning or washing. Steam cleaning of

equipment, if used, will be performed at a temporary decontamination site. The flooring of the

temporary decontamination site will be impermeable to water and large enough to contain the

equipment and the rinsate produced.

If the quantity of water in the pad area exceeds its holding capacity, the water will be drummed

temporarily until analytical results are obtained and the water can be properly disposed of. Steam

cleaning will not be performed over bare ground, but will always be conducted so that rinsate can

be collected and disposed of properly. Wherever applicable, equipment will be disassembled to

permit adequate cleaning of the internal portions.

5.1 Drilling and Large Equipment

The following procedure will be used for decontamination of large pieces of equipment. These

include well casings, auger flights, drill pipes and rods, and those portions of the drill rig that

may stand directly over a boring or well location, or that may come into contact with casing,

auger flights, pipes, or rods.

Establish a decontamination area large enough to contain the equipment and any

decontamination waste

OU3 SOP 7

Rev. No. 0

Date: September 26, 2007

Page 5 of 8

- Place equipment on sawhorse or equivalent, if possible.
- Steam clean the external surfaces and internal surfaces, as applicable, on equipment using high-pressure steam cleaner from an approved water source. If necessary, scrub using brushes and a phosphate-free detergent (e.g., Alconox<sup>TM</sup>), or equivalent laboratory-grade detergent until all visible dirt, grime, grease, oil, loose paint, rust, etc., have been removed.
- Rinse with potable water
- Remove equipment from decontamination pad and allow to air dry
- Record date and time of equipment decontamination

## 5.2 Soil and Groundwater Sampling Equipment

The following procedure will be used to decontaminate sampling equipment such as split-spoon samplers; brass sleeves; continuous core barrels; scoops; hand augers; non-dedicated bailers; submersible pumps, bladder pumps; and other sampling equipment that may come into contact with samples. To minimize decontamination procedures in the field, dedicated equipment will be used wherever feasible:

- Wash and scrub equipment with phosphate-free, laboratory-grade detergent (e.g., Alconox<sup>TM</sup> or equivalent) and off-site distilled water
- Triple-rinse with distilled water
- Air dry
- Wrap in aluminum foil, or store in clean plastic bag or designated casing.

OU3 SOP 7

Rev. No. 0

Date: September 26, 2007

• Record date and time of equipment decontamination

Personnel involved in decontamination activities will wear appropriate protective clothing as

defined in the project-specific health and safety plan.

5.3 Monitoring Equipment

The following procedure will be used to decontaminate monitoring devices such as slug-test

equipment, groundwater elevation and free product thickness measuring devices, and water

quality checking instruments. Note that organic solvents can not be used to decontaminate free

product measuring devices because they will cause damage to the probes. Spray bottles may be

used to store and dispense distilled water.

• Wash equipment with laboratory-grade, phosphate-free detergent (e.g., Alconox<sup>TM</sup> or

equivalent) and distilled water

• Triple-rinse with distilled water

Store in clean plastic bag or storage case.

Record date and time of equipment decontamination

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

All equipment decontamination must be documented in the field logbooks and/or field forms,

including rationales deviations from this SOP. The Field Team Leader or designated QA

reviewer will check and verify that field documentation has been completed per this procedure

and other procedures referenced herein.

To assess the adequacy of decontamination procedures, field rinsate blanks may be collected.

The specific number of rinsate blanks will be defined in a FSP or work plan or by the Project

OU3 SOP 7

Rev. No. 0

Date: September 26, 2007

Page 7 of 8

Manager. In general, at least one field rinsate blank should collected per sampling event or per day.

Rinsate blanks with elevated or detected contaminates will be evaluated by the Project Manager, who will relay the results to the site workers. Such results may be indicative of inadequate decontamination procedures that require corrective actions (e.g., retaining).

#### 7.0 PROCEDURE FOR WASTE DISPOSAL

All decontamination water that has come into contact with contaminated equipment will be handled, labeled, stored and disposed according to SOP 12. Unless otherwise specified in the FSP, waste generated from other sources and classified as non-hazardous waste (e.g., PPE, pastic sheeting, rope and misc. debris) will be disposed into trash receptacles.

#### 8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

U.S. Environmental Protection Agency, RCRA Ground-Water Monitoring: Draft Technical Guidance, November 1992. Page 7-17.

Date: September 26, 2007

OU3 SOP 8 (Rev. 0)

Title: SAMPLE HANDLING AND SHIPPING

APPROVALS:

**TEAM MEMBER** 

**EPA Remedial Project Manager** 

**SOP Author** 

DATE

9/24/07

9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	

OU3 SOP 8 Rev. No. 0 Date: September 26, 2007 Page 1 of 13

1.0 INTRODUCTION

This standard operating procedure (SOP) is based on MWH SOP-09, Sample Handling and

Shipping, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site

OU3. This SOP describes the requirements for sample handling, storage and shipping. The

purpose of this SOP is to define sample management activities as performed from the time of

sample collection to the time they are received by the laboratory.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the

health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen

by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the

lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung

cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases

(ATSDR 2006).

3.0 DEFINITIONS

Chain-of-Custody: An accurate written record of the possession of each sample from the time

of collection in the field to the time the sample is received by the designated analytical

laboratory.

Sample: Physical evidence collected for environmental measuring and monitoring.

For the purposes of this SOP, sample is restricted to solid, aqueous, air, or waste matrices. This

SOP does not cover samples collected for lithologic description nor does it include remote

sensing imagery or photographs (refer to SOP-9 for field documentation procedures).

**Sampler:** The individual who collects environmental samples during fieldwork.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated

with them. This list is not intended to be comprehensive and often additional personnel may be

involved. Project team member information will be included in project-specific plans (e.g., work

plan, field sampling plan (FSP), quality assurance plan, and etc.), and field personnel will always

consult the appropriate documents to determine project-specific roles and responsibilities. In

addition, one person may serve in more than one role on any given project.

Project Manager: The Project Manager is responsible for ensuring that the requirements for

sample management are included in the appropriate project plans. The Project Manager is

responsible for coordinating sample management efforts with input from other key project staff

and applicable government agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and

quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods,

performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader and/or Field Hydrogeologist, Geologist or Engineer: Implements the

sampling program, supervises other sampling personnel, and ensures compliance with SOPs and

QA/QC requirements. Prepares daily logs of field activities.

**Field Technician:** Responsible for sample collection, documentation, packaging, and shipping.

Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks.

5.0 PROCEDURES

5.1 Applicability

The information in this SOP may be used by direct reference or incorporated into project-specific

plans. Deviations or modifications to procedures addressed herein must be brought to the

attention of, and approved by, applicable government agencies.

OU3 SOP 8 Rev. No. 0

Date: September 26, 2007

Page 3 of 13

5.2 Sample Management

Sample Containers: The sample containers to be used will be dependent on the sample matrix

and analyses desired, and are specified in the project FSP. Only certified pre-cleaned sample

containers will be used. Sample containers will be filled with adequate headspace

(approximately 10 percent) for safe handling upon opening, except containers for volatile organic

compound (VOC) analyses, which will be filled completely with no headspace. This no-

headspace requirement applies to both soil and groundwater samples.

Once opened, the containers will be used immediately. If the container is used for any reason in

the field (e.g., screening) and not sent to the laboratory for analysis, it will be discarded. Prior to

discarding the contents of the used container and the container, disposal requirements will be

evaluated. When storing before and after sampling, the containers will remain separate from

solvents and other volatile organic materials. Sample containers with preservatives added by the

laboratory will not be used if held for an extended period on the job site or exposed to extreme

heat conditions. Containers will be kept in a cool, dry place. For preserved samples (except

VOCs), the pH of the sample will be checked following collection of the sample. If the pH is not

at the required level, additional preservative (provided by the laboratory) will be added to the

sample container.

Numbering and Labeling: Refer to OU3 SOP-9.

Custody Seals. Custody seals with the date and initials of the sampler will be used on each

shipping container to ensure custody. The custody seal will be placed on opposites sides of the

cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are

all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of

the liner bag inside of the cooler.

Chain-of-Custody: COC procedures require a written record of the possession of individual

samples from the time of collection through laboratory analyses. A sample is considered to be in

custody if it is:

OU3 SOP 8

Rev. No. 0

Date: September 26, 2007

- In a person's possession
- In view after being in physical possession
- In a secured condition after having been in physical custody
- In a designated secure area, restricted to authorized personnel

The COC record will be used to document the samples taken and the analyses requested. Refer to SOP-9 Attachment 2 for the OU3-specific COC form. Information recorded by field personnel on the COC record will include the following:

- Sample identifier (Index ID)
- Date and time of collection
- Sample matrix
- Preservation
- Type of analyses requested
- Unique COC number
- Lab being shipped to
- Signature of individuals involved in custody transfer (including date and time of transfer)
- Airbill number (if appropriate)
- Any comments regarding individual samples (e.g., organic vapor meter readings, special instructions).

COC records will be placed in a waterproof plastic bag (e.g., Ziploc®), taped to the inside lid of the cooler or placed at the top of the cooler, and transported with the samples. Signed airbills will serve as evidence of custody transfer between the field sampler and courier, as well as between the courier and laboratory. If a carrier service is used to ship the samples (e.g., Federal Express, etc.), custody will remain with the courier until it is relinquished to the laboratory. Upon receiving the sample cooler, a laboratory representative should sign in the receiving box of the COC, thus establishing custody. The sampler will retain copies of the COC record and airbill.

OU3 SOP 8 Rev. No. 0

Date: September 26, 2007

Page 5 of 13

**Sample Preservation/Storage:** The requirements for sample preservation are dependent on the desired analyses and the sample matrix, and are specified in the FSP.

#### 5.3 Sample Shipping

The methods and procedures described in this SOP were developed from these sources:

- 49 CFR 173. Shippers Shippers General Requirements for Shipping. United States Code of Federal Regulations available online at <a href="http://www.gpoaccess.gov/cfr/index.html">http://www.gpoaccess.gov/cfr/index.html</a>
- 49 CFR 178. Specifications for Packaging. United States Code of Federal Regulations available online at <a href="http://www.gpoaccess.gov/cfr/index.html">http://www.gpoaccess.gov/cfr/index.html</a>
- ASTM D 4220. Standard Practice for Preserving and Transporting Soil Samples. American Society for Testing and Materials available online at http://www.astm.org/
- ASTM D 4840. Standard Practice for Sampling Chain-of-Custody Procedures. American Society for Testing and Materials available online at http://www.astm.org/

Procedures for packaging and transporting samples to the laboratory are dependent on the chemical, physical, and hazard properties of the material. The procedures may also be based on an estimation of contaminant concentrations/properties in the samples to be shipped. Samples will be identified as environmental samples, excepted quantities samples, limited quantities samples, or standard hazardous materials. Environmental samples are defined as solid or liquid samples collected for chemical or geotechnical analysis. Excepted quantities involve the shipment of a few milliliters of either an acid or base preservative in an otherwise empty sample container. Limited quantities are restricted amounts of hazardous materials that may be shipped in generic, sturdy containers. Standard hazardous material shipments require the use of stamped/certified containers. All samples will be packaged and shipped or hand delivered to the laboratories the same day of sample collection, unless otherwise specified in the project-specific FSPs.

The following paragraphs describe standard shipping procedures for different types of samples.

Any exceptions to these procedures will be defined in the FSP. It is the responsibility of the

sampler to refer to the U.S. Department of Transportation (DOT)

(http://hazmat.dot.gov/regs/rules.htm) regulations when dealing with a substance not addressed

in this SOP for requirements and limitations associated with the shipment.

Sample Shipping via Commercial Carrier:

Aqueous or Solid Samples: Samples will be packaged and shipped to the laboratories the same

day of sample collection, unless otherwise specified in the FSP and depending on holding time

requirements for individual samples. For aqueous or solid samples that are shipped to the

laboratory via a commercial carrier the following procedures apply:

• Sample labels will be completed and attached to sample containers.

• The samples will be placed upright in a waterproof metal (or equivalent strength

plastic) ice chest or cooler.

• For shipments containing samples for volatile organic analysis, include a trip blank.

• Ice in double Ziploc® bags (to prevent leakage) will be placed around, among, and on

top of the sample bottles. Enough ice will be used so that the samples will be chilled

and maintained at 4°C ± 2°C during transport to the laboratory. Dry ice or blue ice

will not be used.

• To prevent the sample containers from shifting inside the cooler, the remaining space

in the cooler will be filled with inert cushioning material, such as shipping peanuts,

additional bubble pack, or cardboard dividers, such that the sample containers remain

upright and do not break.

Tape shut the cooler's drain plug

OU3 SOP 8

Rev. No. 0

Date: September 26, 2007

• The original copy of the completed COC form will be placed in a waterproof plastic

bag and taped to the inside of the cooler lid or placed at the top of the cooler.

The lid will be secured by wrapping strapping tape completely around the cooler in

two locations.

• Mark the cooler with arrow labels indicating the proper upright position of the cooler.

• Custody seals consisting of security tape with the date and initials of the sampler will

be used on each shipping container to ensure custody. Two signed custody seals will

be placed on the cooler, one on the front and one on the back.

A copy of the COC record and the signed air bill will be retained for the project files.

• Affix a label containing the name and address of the shipper to the outside of the

cooler

Hand-Delivered Samples: For aqueous or solid samples that will be hand carried to the

laboratory, the same procedures apply.

Excepted Quantities: Usually, corrosive preservatives (e.g., hydrochloric acid, sulfuric acid,

nitric acid, or sodium hydroxide) are added to otherwise empty sample bottles by the analytical

laboratory prior to shipment to field sites. However, if there is an occasion whereby personnel

are required to ship bottles with these undiluted acids or bases, the containers will be shipped in

the following manner:

1. Each individual sample container will have not more than 30 milliliters of

preservative.

- 2. Collectively, the preservative in these individual containers will not exceed a volume of 500 milliliters in the same outer box or package.
- 3. Despite the small quantities, only chemically compatible material may be placed in the same outer box, (e.g., sodium hydroxide, a base, must be packaged separately from the acids).
- 4. Federal Express will transport nitric acid only in concentrations of 40 percent or less.
- 5. A "Dangerous Goods in Excepted Quantities" label will be affixed to the outside of the outer box or container. Information required on the label includes:
  - Signature of Shipper
  - Title of Shipper
  - Date
  - Name and Address of Shipper
  - Check of Applicable Hazard Class
  - Listing of UN Numbers for Materials in Hazard Classes

Limited Quantities: Occasionally, it may become necessary to ship known hazardous materials, such as pure or floating product. DOT regulations permit the shipment of many hazardous materials in "sturdy" packages, such as an ice chest or cardboard box (not a specially constructed and certified container), provided the following conditions are met:

- Each sample bottle is placed in a plastic bag, and the bag is sealed. Each VOC vial
  will be placed in a sealable bag. As much air as possible is squeezed from the bag
  before sealing. Bags may be sealed with evidence tape for additional security.
- 2. Or each bottle is placed in a separate paint can, the paint can is filled with vermiculite, and the lid is affixed to the can. The lid must be sealed with metal clips, filament, or evidence tape. If clips are used, the manufacturer typically recommends six clips.

OU3 SOP 8 Rev. No. 0

Date: September 26, 2007

Page 9 of 13

- 3. The cans are placed upright in a cooler that has had the drain plug taped shut inside and outside, and the cooler is lined with a large plastic bag. Approximately 1 inch of adsorbent material sufficient to retain any liquid that may be spilled, is placed in the bottom of the liner. Only containers having chemically compatible material may be packaged in each cooler or other outer container.
- 4. The COC record is sealed inside a plastic bag and placed inside the cooler. The sampler retains one copy of the COC record. The laboratory will be notified if the sample is suspected of containing any substance for which the laboratory personnel should take safety precautions.
- 5. The cooler is shut and sealed with strapping tape (filament type) around both ends. Two signed custody seals will be placed on the cooler, one on the front and one on the back. Additional seals may be used if the sampler and/or shipper consider more seals to be necessary. Wide, clear tape will be placed over the seals to ensure against accidental breakage.
- 6. The following markings are placed on the side of the cooler:
  - Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA <u>Dangerous Goods Regulations [DGR]</u>)
  - UN Number (Column A, List of Dangerous Goods, Section 4, IATA <u>DGR</u>)
  - Shipper's name and address
  - Consignee's name and address
  - The words "LIMITED QUANTITY"
  - Hazard Labels (Column E, List of Dangerous Goods, Section 4, IATA <u>DGR</u>)
  - Two Orientation (Arrow) labels placed on opposite sides.
- 7. The Airbill/Declaration of Dangerous Goods form is completed as follows:

OU3 SOP 8 Rev. No. 0

Date: September 26, 2007

- Shipper's name and address
- Consignee's name and address
- Services, Delivery & Special Handling Instructions
- Cross out "Cargo Aircraft Only" in the Transport Details Box
- Cross out "Radioactive" under Shipment Type
- Nature and Quantity of Dangerous Goods
  - Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA <u>DGR</u>)
  - Class or Division (Column C, List of Dangerous Goods, Section 4, IATA <u>DGR</u>)
  - UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
  - Packing Group (Column F, List of Dangerous Goods, Section 4, IATA <u>DGR</u>)
  - Subsidiary Risk, if any (Column D, List of Dangerous Goods, Section 4, IATA <u>DGR</u>)
  - Quantity and type of packing (number and type of containers: for example, "3 plastic boxes", and the quantity per container, "2 L", is noted as "3 Plastic boxes X 2 L" This refers to 3 plastic boxes (coolers are referred to as plastic boxes) with 2 liters in each box.
  - Packing Instructions (Column G, List of Dangerous Goods, Section 4, IATA DGR).
  - Note: Only those Packing Instructions in Column G that begin
    with the letter "Y" may be used. These refer specifically to the
    Limited Quantity provisions.
  - Authorization (Write in the words Limited Quantity)
  - Emergency Telephone Number (List 800-535-5053. This is the number for INFOTRAC.)
  - Printed Name and Title, Place and Date, Signature.

OU3 SOP 8 Rev. No. 0 Date: September 26, 2007

Page 11 of 13

**Standard Hazardous Materials:** Shipment of standard hazardous materials presents the most difficulty and expense. However, there may be occasion whereby a hazardous material cannot be shipped under the Limited Quantity provisions, (e.g., where there is no Packing Instruction in Column G, List of Dangerous Goods, IATA <u>Dangerous Goods Regulations</u>, that is preceded by the letter "Y").

In such cases, the general instructions noted above but for non-Limited Quantity materials will apply, with one important difference: standard hazardous materials shipment requires the use of certified outer shipping containers. These containers have undergone rigid testing and are, therefore, designated by a "UN" stamp on the outside, usually along the bottom of a container's side. The UN stamp is also accompanied by codes specifying container type, packing group rating, gross mass, density, test pressure, year of manufacturer, state of manufacturer, and manufacturer code name. The transport of lithium batteries in Hermit Data Loggers is an example of a standard hazardous material where only a designated outer shipping container may be used.

# 5.4 Holding Times

The holding times for samples will depend on the analysis and the sample matrix. Refer to the FSP for holding times requirements.

#### 6.0 QUALITY ASSURANCE AND QUALITY CONTROL

All sample shipments must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that handling and shipment documentation has been completed per this procedure and other procedures referenced herein.

#### 7.0 DECONTAMINATION

All shipment coolers shall be maintained clean of sampled material to avoid exposure during shipment. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

# 8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors, Draft, Appendix D, April 1980.

OU3 SOP 8 Rev. No. 0 Date: September 26, 2007

Page 13 of 13

Date: June 30, 2008

OU3 SOP 9 (Rev. 4)

Title: FIELD DOCUMENTATION

APPROVALS:

TEAM MEMBER

EPA Remedial Project Manager

SOP Author

-SIGNATURE/TITLE

DATE

7/1/08

	1	
Revision Number	Date	Reason for Revision
0	09/26/2007	
1	10/5/2007	Add section for "Corrections and Modifications" and

0	09/26/2007	
1	10/5/2007	Add section for "Corrections and Modifications" and Field Modification Approval form (Attachment 3) Update Labeling section and COC (Attachment 2) to reflect non-asbestos analysis and container details Update FSDS forms (Attachment 1) based on field team input
2	02/22/2008	Incorporate changes to FSDS forms (Attachment 1)     based on field input     Remove OU3 phase specificity in SOP text
3	05/29/2008	Incorporate changes to FSDS forms (Attachment 1)     based on field input
4	06/30/2008	Update Attachment 1 with all OU3 FSDS forms (including those used in Phase I and Phase II)     Remove OU3 phase specificity in Attachments

OU3 SOP 9 Rev. No. 4

Date: June 30, 2008 Page 1 of 11

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is a general guidance document for the required

documentation to be completed by field personnel during field investigations. This SOP is based

on MWH SOP-04, Field Documentation, Revision 1.0, March 2006, modified for use at the

Libby Mine Site. Documentation in the form of field logbooks, reports, and forms shall be

completed for every activity in the field. Records shall be maintained on a daily basis as the

work progresses. All field documentation shall be accurate and legible because it is deliverable

to the client as potentially a legal document.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the

site health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be

seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in

the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of

lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory

diseases (ATSDR 2006). All personnel engaged in soil sampling must follow health and safety

protocols described in the health and safety plan.

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated

with them. This list is not intended to be comprehensive and often, additional personnel may be

involved. Project team member information shall be included in project-specific plans (e.g.,

work plan, field sampling plan, quality assurance plan, etc.), and field personnel shall always

consult the appropriate documents to determine project-specific roles and responsibilities. In

addition, one person may serve in more than one role on any given project.

Project Manager: Selects project-specific field documentation with input from other key

project staff.

OU3 SOP 9

Rev. No. 4 Date: June 30, 2008

Dana 2 af 11

Page 2 of 11

Quality Control Manager: Overall management and responsibility for quality assurance and

quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods,

performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements

the sampling program, supervises other sampling personnel, and ensures compliance with SOPs

and QA/QC requirements. Prepares daily logs of field activities.

Field Technician (or other designated personnel): Assists the FTL and/or field geologist,

hydrogeologist, or engineer in the implementation of field tasks and field documentation.

Field Sample/Data Manager: Responsible for proper handling and shipping of all samples

collected by the field crew, electronic data entry of field sample data sheet (FSDS) and chain-of-

custody (COC) forms, and scanning/posting of field documentation PDFs (FSDS, COC, field

logbooks, digital photographs) to a dedicated FTP site.

4.0 FIELD DOCUMENTATION PROCEDURES

Field documentation serves as the primary foundation for all field data collected that will be used

to evaluate the project site. There are two main forms of field documentation – field logbooks

and FSDS forms. All field documentation shall be accurate, legible and written in indelible

black or blue ink. Absolutely no pencils or erasures shall be used. Incorrect entries in the FSDS

forms or field logbooks will be corrected by crossing out the incorrect entry with one line, the

individual making the correction will initial and date next to the correction.

4.1 Field Logbooks

The field logbook shall be a bound, weatherproof book with numbered pages, and shall serve

primarily as a daily log of the activities carried out during the fieldwork. All entries shall be

made in indelible black or blue ink. A field logbook shall be completed for each operation

undertaken during the field tasks. To further assist in the organization of the field log books, the

project name and the date shall be recorded on top of each page along with the significant

OU3 SOP 9 Rev. No. 4

activity description (e.g., surface sample or soil boring number). All original field documentation shall be retained in the project files.

Skipped pages or blank sections at the end of a field log book page shall be crossed out with an "X" covering the entire page or blank section; "No Further Entries," initials, and date shall be written by the person crossing out the blank section or page. The responsible field team member shall write his/her signature, date, and time after the day's last entry.

Field activities vary from project to project; however, the concept and general information that shall be recorded are similar. The descriptions of field data documentation given below serve as an outline; individual activities may vary in documentation requirements. A detailed description of two basic example logbooks, suitable for documentation of field activities, is given below. These field logbooks include the FTL logbook and the field geologist/sampling team logbook.

FTL Logbook: The FTL's responsibilities include the general supervision, support, assistance, and coordination of the various field activities. As a result, a large portion of the FTL's day is spent rotating between operations in a supervisory mode. Records of the FTL's activities, as well as a summary of the field team(s) activities, shall be maintained in a logbook. The FTL's logbook shall be used to fill out daily/weekly reports and daily quality control reports (DQCRs), and therefore, shall contain all required information. Entries shall be preceded with time in military units for each observation. Items to be documented include:

- Record of tailgate meetings
- Personnel and subcontractors on job site and time spent on the site
- Field operations and personnel assigned to these activities
- Site visitors
- Log of FTL's activities: time spent supervising each operation and summary of daily operations as provided by field team members
- Problems encountered and related corrective actions
- Deviations from the sampling plan and reasons for the deviations
- Records of communications; discussions of job-related activities with the client,
   subcontractor, field team members, and project manager

OU3 SOP 9 Rev. No. 4 Date: June 30, 2008 Page 4 of 11

- Information on addresses and contacts
- Record of invoices signed and other billing information
- Field observations

**Field Geologist/Sampling Team Logbook:** The field geologist or sampling team leader shall be responsible for recording the following information in a logbook:

- Health and Safety Activities
  - Calibration records for health and safety equipment (e.g., type of PID, calibration gas used, associated readings, noise dosimeters, etc.)
  - Personnel contamination prevention and decontamination procedures
  - Record of daily tailgate safety meetings
- Weather
- Calibration of field equipment
- Equipment decontamination procedures
- Personnel and subcontractors on job site and time spent on the site
- Station identifier
- Sampling activities
  - Sample location (sketch)
  - Equipment used
  - Names of samplers
  - Date and time of sample collection
  - Sample interval
  - Number of samples collected
  - Analyses to be performed on collected samples
- Disposal of contaminated wastes (e.g., PPE, paper towels, Visqueen, etc.)
- Field observations
- Problems encountered and corrective action taken
- Deviations from the sampling plan and reason for the deviations
- · Site visitors

## 4.2 Field Sample Documentation

Sample Labels: A unique sample identification label shall be affixed to all sample containers. All samples will be labeled in a clear, precise way for proper identification in the field and for tracking in the laboratory. At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number, referred to as the Index ID. The Index ID for all samples collected as part of OU3 sampling activities will have a two-character prefix specific to the sampling Phase (e.g., Phase 1 samples will have a "P1" prefix, P1-12345) as specified in the applicable SAP. Index ID labels will be ½ inch x 1 ¾ inch in size and pre-printed for use in the field. For each Index ID, multiple labels will be printed to allow for multiple containers of the same sample (i.e., for different analyses).

Index ID Label Example:

P1-12345

Each collection container will be labeled with a container label that enables the field team member to record the container specific details, such as the method of sample preparation (e.g., filtered/unfiltered), method of preservation, and the analytical methods that will be requested. Container labels will be 2 inch x 4 inch in size and pre-printed for use in the field. Any container-specific information shall be written in indelible ink.

Container Label Example:

Date/Time:
Media (circle one): AQ SO AA BK DB TC For AQ, Filtered? (circle one): Yes No Container:
Preservation: Analyses:
Analyses:

Media acronyms: AQ – aqueous media, SO – solid media, AA – ambient air, BK – tree bark, DB – organic debris, TC – tree age core

OU3 SOP 9 Rev. No. 4 Date: June 30, 2008 Page 6 of 11

After labels have been affixed to the sample container, the labels will be covered with clear

packaging tape to ensure permanence during shipping.

Any unused Index ID labels should be crossed out to avoid the possibility of using unused labels

for a different sample.

Field Sample Data Sheet (FSDS) Forms: Data regarding each sample collected as part of the

OU3 sampling will be documented using Libby-specific FSDS forms (provided as Attachment

1). These FSDS forms are media-specific and designed to facilitate data entry of station

location, sample details, and field measurements needed for the OU3 investigation.

In the field, one field team member will be responsible for recording all sample details onto the

appropriate FSDS form. At the time of sample labeling, one Index ID label will be affixed to the

FSDS form in the appropriate field. All written entries on the FSDS form shall be accurate,

legible and written in indelible black or blue ink.

Once the FSDS form is complete, written entries will be checked by a second field team

member. These two field team members will initial the bottom of the FSDS form in the

appropriate field to document who performed the written data entry and who performed the QC

check of the FSDS form.

On a weekly basis (or more frequently as conditions permit), information from the hard copy

FSDS form will be manually entered into a field-specific OU3 database using electronic data

entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of

all data entry will be completed by the FTL or their designate. The Field Sample/Data Manager

and the FTL will initial in the appropriate field on the paper FSDS form to document who

performed the data entry into the database and who performed the QC check.

4.3 Photologs

Photologs are often used in the field to document site conditions and sample location

characteristics. While photographs may not always be required, they shall be used wherever

applicable to show existing site conditions at a particular time and stage of the investigation or

related site activity. Photolog information shall include:

• station location identifier

Index ID (if applicable)

date and time of photo

direction/orientation of the photo

description of what the photo is intended to show

An engineer's scale or tape shall be included in any photographs where scale is necessary. Any

wasted frames or images in a roll of film or sequence of digital images shall be so noted in the

field logbook.

4.4 Chain-of-Custody Records

Custody Seals: Custody seals with the date and initials of the sampler will be used on each

shipping container to ensure custody. The custody seal will be placed on opposites sides of the

cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are

all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of

the liner bag inside of the cooler.

**Chain-of-Custody Forms**: COC procedures allow for the tracking of possession and handling

of individual samples from the time of field collection through to laboratory analysis.

Documentation of custody is accomplished through a COC form that lists each sample and the

individuals responsible for sample collection and shipment, sample preparation, and receipt by

the analytical laboratory. The COC form also documents the analyses requested for each

sample. Whenever a change of custody takes place, both parties will sign and date the COC

form, with the relinquishing party retaining a copy of the form. The party that accepts custody

will inspect the COC form and all accompanying documentation to ensure that the information is

complete and accurate. Any discrepancies will be noted on the COC form. Shipping receipts

shall be signed and filed as evidence of custody transfer between field sampler(s), courier, and

laboratory.

OU3 SOP 9

Rev. No. 4

Date: June 30, 2008

Page 8 of 11

Attachment 2 provides an example of the COC form that will be used for all samples collected as

part of OU3 sampling. This form will be printed as a carbonless triplicate form to facilitate

retention of COC copies by relinquishing parties. As seen, the COC form includes the following

information:

sample identifier (Index ID)

date and time of collection

method of sample preparation and preservation

number of sample containers

analyses requested

shipping arrangements and airbill number, as applicable

recipient laboratories

signatures of parties relinquishing and receiving the sample

On a daily basis, the Field Sample/Data Manager will package samples for shipping, complete

hard copy COC forms, and ship all samples as outlined in SOP No. 8. On a daily basis,

information from the hard copy COC form necessary for sample tracking will be manually

entered into a field-specific OU3 database using electronic data entry screens by the Field

Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be

completed by the FTL or their designate.

5.0 FIELD DATA TRANSMITTAL

Copies of all FSDS forms, COC forms, and field log books will be scanned and posted in

portable document format (PDF) to a project-specific file transfer protocol (FTP) site daily. This

FTP site will have controlled access (i.e., user name and password are required) to ensure data

access is limited to appropriate project-related personnel. File names for scanned FSDS forms,

COC forms, and field log books will include the sample date in the format YYYYMMDD to

facilitate document organization (e.g., FSDS\_20070831.pdf).

OU3 SOP 9 Rev. No. 4

Date: June 30, 2008

Page 9 of 11

Electronic copies of all digital photographs will also be posted weekly (or more frequently as conditions permit) to the project-specific FTP site. File names for digital photographs will include the station identifier, the sample date, and photograph identifier (e.g., ST-1\_20070831\_12459.tif).

A copy of the field-specific OU3 database will be posted to the project-specific FTP site on a weekly basis (or more frequently as conditions permit). The field-specific OU3 database posted to the FTP site will include the post date in the file name (e.g., FieldOU3DB 20070831.mdb).

#### 6.0 CORRECTIONS AND MODIFICATIONS

#### 6.1 Field Deviations and Modifications

It is recognized that deviations and modifications from the standard operating procedures may be necessary based on site conditions. Any requested field modifications will be submitted by Robert Marriam (Remedium Group, Inc. - W.R. Grace contractor) to Bonita Lavelle (EPA Region 8 - Remedial Project Manager) for review and approval. All modification requests will be recorded in a Field Modification Approval Form (see Attachment 3).

#### 6.2 Corrections to Hard Copy Forms

If an error is identified on an FSDS or COC form <u>prior to entry</u> into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. Data entry into the field-specific OU3 database and scanning/posting of the hard copy forms should proceed following the data entry procedures described above.

If an error is identified on an FSDS or COC form <u>after entry</u> into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. The corrected form should be scanned and posted to the project-specific FTP site. File names for corrected FSDS forms will include the Index ID of the corrected sample to

OU3 SOP 9 Rev. No. 4 Date: June 30, 2008 Page 10 of 11

facilitate document organization (e.g., FSDS\_C\_P1-12345.pdf). File names for corrected COC

forms will include the COC ID of the corrected COC form to facilitate document organization

(e.g., COC\_C\_OU3-36512.pdf). Necessary data corrections will be made to the master OU3

database by the database manager.

If changes are made to a COC form, the analytical laboratory should be provided with a

corrected COC form.

7.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

RCRA Ground-Water Monitoring: Draft Technical Guidance, November 1992.

OU3 SOP 9 Rev. No. 4

Date: June 30, 2008

Page 11 of 11

# **ATTACHMENT 1**

OU3 FIELD SAMPLE DATA SHEET (FSDS) FORMS

Sheet No.:	SWS2
	SED2

# LIBBY OU3 PHASE II FIELD SAMPLE DATA SHEET SURFACE WATER AND SEDIMENT

Station II		):		<del>-</del>						
GPS Coo For New Sampling	Stations		X co	Zone 11 Nort ord: lers Initials:		Y coor	d:	eters		
WATER (	ידו ואנום	Y PARAM	FTFRS (	if applicable)						
Time Meas (hh:mm)	ured	Temp. (°C)	pΗ	Specific Conduct (mS/cm Auto-comp @	f f	Diss. (mg/l	-	ORP (mV)		Turbidity (NTU)
SAMPLE	COLLE	CTION		<u></u>						
Index ID	A	AFFIX LABEL	HERE	Sampling Tim Sample Type:	Field	Sample		Grab # of Comp	or posites: Depth: To	if applicable): Composite  pp (In) ot (in)
Index ID	А	AFFIX LABEL	HERE	Sampling Tim Sample Type:	ne: SI MS FB	P FI MSD TB	PE EB	Grab # of Comp	or posites: Depth: To	if applicable): Composite  pp (in) ot (in)
Index ID	A	FFIX LABEL .	HERE	Sampling Tim Sample Type: Media: Surfa	MS FB	P FI MSD TB	PE EB	Grab # of Comp	or osites: Depth: To	if applicable): Composite  op (in) ot (in)
Index ID	А	FFIX LABEL I	HERE	Sampling Tim Sample Type: Media: Surfa	MS FB	P FI MSD TB	PE EB	Grab # of Comp	or osites: Depth: To	f applicable): Composite op (in) ot (in)
COMMEN	NTS			1				<u> </u>		
Note:	FS TB FB	Field Sampl Trip Blank S Field Blank S	ample	•	ike Sample	e lank Sample	3	MSD Matrix Sp	olicate Sample ike Duplicate S nce Evaluation	•
Field Da	nta Entere	ed by:			Fleid E	ntries (	hecked	l by:	<del></del>	
	Dat	tabase Entry:			Date	abase QC	<del></del>			7

Sheet	No.:	AA2-	

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2 STATIONARY AMBIENT AIR MONITOR

Field Logbook No:			ONARY AI Page No:						if GPS information
Station ID:								Deen recoi	dea previously
GPS Coordinate System X coord:Sampling Team:	n: <u>UTM Zo</u>	one 11 North	h, NAD83 datum, m coord:	neters				r	m
Data Item		Casse	ette 1		Casse	ette 2		Casse	ette 3
index iD	,	AFFIX LABE	EL HERE	AFFIX LABEL HERE				AFFIX LABEL HERE	
Sample Height (ft)						<u></u>			·
Location Description									
Field QC Type (circle)	FD-(field	i sample) i dup) Parent ID:	FB-(field blank)	FD-(field	sample) I dup) Parent ID:	FB-(field blank)	FD-(field	i sample) i dup) Parent ID:	FB-(field blank)
Matrix Type		Outdo	oor		Outd	loor		Outd	oor
Flow Meter Type	<del>                                     </del>	Rotam	eter	ļ	Rotan	neter	<b> </b>	Rotameter	
Archive blank (circle)	<del> </del> `	Yes	No	<u> </u>	Yes	No		Yes	No
Pump ID Number	<u> </u>								
Flow Meter ID Number	<u></u>						<u> </u>		
Start Date (mm/dd/yy)	<u> </u>								
Start Time (hh:mm)									
Start Counter									
Daily Flow Check:	Check1	Time	Flow	Check1	Time	Flow	Check1	Time	Flow
	Check2			Check2			Check2		
Record time (hh:mm) and flow rate (L/min)	Check3			Check3			Check3		
in fields provided	Check4			Check4			Check4		
Stop Date (mm/dd/yy)									<del></del>
Stop Time (hh:mm)									
Stop Counter									
Pump fault? (circle)	``	Yes	No	\	⁄es	No	,	Yes	No
Stop Flow (L/min)									
Field Comments									
Cassette Lot Number:									

For Data Entry Completion (Provide Initials)	Completed by:	QC by:

Validated By (Provide initials):

Entered By (Provide initials):

			She	et No.: GW2
LII	BBY OU3 FIELD SA	AMPLE DATA S	SHEET (FSD	OS) rev3
	GI	ROUNDWATER		☐ Check box if GPS information
Field Logbook No:	Pag	ge No:		has been recorded previously
Station ID:	Station Alias:	-	Sampling	Date:
·	n: <u>UTM Zone 11 North, NAD83 da</u>	tum, meters		
	Y coord:			
	Sampler Initia	ıls:		
Station Comments:				
	MP) of Well: MP U		Calibration:	
1	.): Filter Pack Interval (		Daily Verificat	ion Weekly Date:
	Sample Intake Depth (ft	BMP):	VOA Vial pH:	Other pH:
Purge Method:				<del></del>
☐ Dedicated Submersible	le (SP) Portable Bladder (B	BP)		Multiplication Factors:
Portable Submersible	(SP) Dedicated Bailer (B	l)		1" = 0.04 2" = 0.16 3" = 0.37 4" = 0.65
Dedicated Bladder (SF	P) Disposable Bailer (	B)	1	5" = 1.47
Purge:			_	
1	MP): Total De			
	_			: 4X:
Water Level (ft BMP) at E	nd of Purge:	_ Total Depth (ft BMP) a	at End of Purge:	
Time Temp. (hh:mm) (°C)	pH	Diss. O <sub>2</sub> Turbidity (mg/L) (NTU)		low Rate (gal/min) Comments
(111.11117)	(marchi@25 c) (mv)	(mg/L) (NTU)	(94.)	gairmin
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Final Parameters	<u>l</u>	<u></u>		
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Data Item	Sample 1	Samı	ple 2	Sample 3
Index ID	AFFIX LABEL HERE	AFFIX LAI	BEL HERE	AFFIX LABEL HERE
Field QC Type	FS SP FD MS MS	D FS SP FD	MS MSD	FS SP FD MS MSD
(circle one):	DE ED ED ED ED ED ED ED			
Field Comments:				
Cooler:				
Entered by (Provide initia	als):	Validated by (Pr	ovide initials):	
<b>TB</b> Tri	ip Blank Sample MS M	ield Split Sample latrix Spike Sample quipment Decon Blank Sample	MSD Mat	d Duplicate Sample rix Spike Duplicate Sample formance Evaluation Sample

Completed by

QC by

For Data Entry Completion (Provide Initials)

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2 FOREST SOIL AND TREE BARK

Field Logbook No:	Page No:			
AA		ate:		
<b>GPS Coordinate System</b>	1: UTM Zone 11 North, NAD83 datum, meters			
X coord:	Y coord:	<del></del>	Elevation:	m
Sampling Team:	Sampler Initials:			
Station Comments:				
TREE BARK SAMPLES				· · · · · · · · · · · · · · · · · · ·
Index ID:	Field QC Type (circle one):	Sample	Tree Species:	Age Core
	FS (field sample)	Area (cm²):		_ Collected?
	FD (field duplicate)			(circle one):
	For FD, Parent ID:		Collection Height (ft)	: Y N
Index ID:	Field QC Type (circle one):	Sample		
	FS (field sample)	Area (cm²):	Diameter* (in):	
	FD (field duplicate)			
	For FD, Parent ID:	_		
*Measured with "D-tape"	·		·	<del></del>
•				
FOREST SOIL SAMPLES Index ID:	Field QC Type (circle one):	Bulk Sa	oil Description	Organic Debris
midex ib.	FS (field sample)	Depth (in)	Sample Type:	Collected?
	FD (field duplicate)	1 ' ' '		(circle one):
	For FD, Parent ID:	Start:		Y N
Index ID:	Field QC Type (circle one):	End:		Organic Debris
muex ID.	FS (field sample)		oil Description	Collected?
	FD (field duplicate)	Depth (in) Start:	Sample Type:  Grab Composite	(circle one):
	For FD, Parent ID:	End:	- I ' I	ΥN
Index ID:	Field QC Type (circle one):		oil Description	Organic Debris
mack is:	FS (field sample)	Depth (in)	Sample Type:	Collected?
	FD (field duplicate)	Start:	Grab Composite	(circle one):
	For FD, Parent ID:	End:	# of Comp.:	Y N
Field Comments:	1	1		
Entered by (Provide initials)	);	Validated by (F	Provide initials):	······································
	<del> </del>	. <del> </del>		

For Data Entry Completion (Provide Initials)	Completed by	QC by

Sheet No.: MS2-

# LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev1 SOIL-LIKE MATERIALS

Field Logbook No:	Page No:	
Station ID:	Sampling Date:	
GPS Coordinate System: <u>uтм</u>	Zone 11 North, NAD83 datum, meters	
Sampling Team:	Sampler Initials:	
Station Comments:		

Data Item	Sample 1	Sample 2	Sample 3						
Index ID	AFFIX LABEL HERE	AFFIX LABEL HERE	AFFIX LABEL HERE						
Matrix (circle one):	Surface Soil Tailings Waste Rock Roadway Other	Surface Soil Tailings Waste Rock Roadway Other	Surface Soil Tailings Waste Rock Roadway Other						
Sample Time (hh:mm)									
Sample Type (circle one):	Grab Composite	Grab Composite	Grab Composite						
	# of Comp:	# of Comp:	# of Comp:						
Sample Depth	Start Depth (in):	Start Depth (in):	Start Depth (in):						
	End Depth (in):	End Depth (in):	End Depth (in):						
Field QC Type	FS (field sample)	FS (field sample)	FS (field sample)						
(circle one):	FD (field duplicate) For FD, Parent ID:	FD (field duplicate) For FD, Parent ID:	FD (field duplicate) For FD, Parent ID:						
	TB (trip blank) Cooler:	TB (trip blank) Cooler:	TB (trip blank) Cooler:						
	PE (perf. eval.) ID:	PE (perf. eval.) ID:	PE (perf. eval.) ID:						
Transect Start Location or	X coord:m	X coord:m	X coord:m						
Grab Sample Location	Y coord:m	Y coord:m	Y coord:m						
	Elevation:m	Elevation:m	Elevation:m						
Transect End Location	X coord:m	X coord:m	X coord:m						
	Y coord:m	Y coord:m	Y coord:m						
Field Comments:	Elevation:m	Elevation:m	Elevation: m						
ried Comments.									
Cooler:									
Entered by (Provide	initials):	Validated by (Provide initials	)·						

For Data Entry Completion (Provide Initials)	Completed by	QC by

# **ATTACHMENT 2**

**OU3 CHAIN OF CUSTODY FORM** 

LIBBY OU3	– CHA	IN-OF	-CU	STOD	ΥI	RE	C	ORD	/R	EC	บเ	ES	ST	F	OF	R A	۱N	IA	LY	'S	IS							С	0	C 1	٧o	<b>-</b>					_
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METHOD OF SHIP	THOD OF SHIPMENT:									CARRIER/WAYBILL NO.:													DESTINATION:												_		
	SAM	IPLE\$					-	<del></del>							_					A٨	IAL`	/SI	S R	EQI	JES	T			_								
				# ¥	Γ		As	bestos Non-Asbestos (a)														$\Box$															
Index ID	Date	Time	Media*	Air Volume (L) or Tree Bark Sample Area (cm²)	Filtered	Archive	TEM-ISO 10312 (b,c)	лгм (d)	TAL Metals+Boron	Mercury	тос	30C	Paste pH	Fluoride	Chloride, Sulfate	Total Phosphorus	Syanide	VРН	ЪН	OPP Pesticides	Chlorinated Pesticides	Herbicides	ross	5000	TOS TSS Nitrite Alkalinity	Ammonia Nitrate TKN	Orthophosphate	Sadiochemistry	Sadiom Transum	Aardnese	Tardness			Age core (e)	R	Remark	
																				Ĭ	Ĭ	T		Ι					Ĭ	I	Ī	ightharpoons	$\Box$				
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* Media: AQ - Aqueous SO -	- Solid AA – Am	bient Air BK -	Tree Bark	DB - Organ	ic Del	nis (D	uff) 1	C - Tree	Age (	core								<del></del>							<u> </u>			-									

Notes -(a) Method, container, and preservation details are provided in the attached tables
(b) With Libby-specific modifications. See applicable O3 SAP for counting and stopping rules
(c) For tree bark, preparation by TREE-LIBBY-OU3. For organic debris (duff), preparation by DUFF-LIBBY-OU3.

DISTRIBUTION: PINK: Field Copy YELLOW: Laboratory Copy WHITE: Return to Originator

<sup>(</sup>d) Preparation by ISSI-LIBBY-01 and analysis by SRC-LIBBY-01 (PLM-Grav) and SRC-LIBBY-03 (PLM-VE) (e) In accordance with procedures in Phipps (1985).

# **ATTACHMENT 3**

**OU3 FIELD MODIFICATION APPROVAL FORM** 

LIBBY OU3	- CHA	IN-OF	-cu	STOD	ΥI	RE	CC	RE	)/R	EC	วบ	ES	ST	F	OR	A	N	٩L	YS	is							C			_				<del></del>		
ENTEDED BY (Size	naturo):									JE/	~T #		NIA.	CE	D.		_			-							_				-		OF:			
ENTERED BY (Sig	nature): _							- '	RU	J⊏(	ווכ	VIA	MA	GEI	Κ													υA	\IE	·-		—-				
METHOD OF SHIP	MENT: _							_ (	CAR	RIE	R/V	۷A۱	YBI	LL I	NO.	:						DE	ST	INA.	TIO	N: .								<del></del> _		
	SAM	ANALYSIS REQUEST  Asbestos Non-Asbestos (a)																																		
		1		3ark	1		As	bestos	4									_	Non-/	Asbes	tos (	a)						_		,						
Index ID	Date	Time	Media*	Air Volume (L) or Tree Bark Sample Area (cm²)	Filtered	Archive	TEM-ISO 10312 (b,c)	PLM (d)	TAL Metals+Boron	Mercury	TOC	DOC	Paste pH	Fluoride	Chloride, Sulfate	I otal Phosphorus	VPH	Hdi	OPP Pesticides	Chlorinated Pesticides	Herbicides	PCBs	SVOCS	TDS. TSS. Nitrite. Alkalinity	Ammonia, Nitrate, TKN	Orthophosphate	Radiochemistry	Radium, Uranium	Hardness			Age core (e)	Rei	marks		
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(d) Preparation by ISSI-LIBBY-01 and analysis by SRC-LIBBY-01 (PLM-Grav) and SRC-LIBBY-03 (PLM-VE) (e) In accordance with procedures in Phipps (1985).

Notes — (d) (d) (e) Method, container, and preservation details are provided in the attached tables (e) (f) (f) With Libby-specific modifications. See applicable O3 SAP for counting and stopping rules (e) (f) For tree bark, preparation by TREE-LIBBY-OU3. For organic debris (duff), preparation by DUFF-LIBBY-OU3.

DISTRIBUTION: PINK: Field Copy YELLOW: Laboratory Copy WHITE: Return to Originator

Date: September 26, 2007

OU3 SOP 10 (Rev. 0)

Title: FIELD EQUIPMENT CALIBRATION

APPROVALS:

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Remedial Project Manager

SOP Author

9/24/07

9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	-

OU3 SOP 10 Rev. No. 0 Date: September 26, 2007 Page 1 of 7

1.0 INTRODUCTION

This standard operating procedure (SOP) provides general guidelines on calibration and

operating procedures for typical field equipment. This SOP is based on MWH SOP-01, Field

Equipment Calibration, Revision 1.0, March 2004, modified for use at the Libby Asbestos

Superfund Site OU3. Field monitoring instruments are used when data quality objectives specify

screening level analytical support. These data are collected for on site, real-time measurements,

evaluation of existing conditions, refinement of sampling locations, and health and safety

evaluations. Field measurements are generally used to refine sampling programs and to estimate

the extent of contamination at the site. This type of support also provides real-time data for

health and safety purposes.

The purpose of this SOP is to define the calibration and operating procedures for equipment used

for field monitoring.

2.0 DEFINITIONS

Conductivity: Is a measure of the quantity of electricity transferred across a unit area, per unit

potential gradient, per unit time. Conductivity is measured by dipping a probe directly into the

water source or into a separate sample aliquot.

**Dissolved Oxygen (DO):** Is a measure of the quantity of oxygen dissolved in water. DO data is

collected in the field using direct measurement probes.

**pH:** Is measured directly using a probe and is the acidity or alkalinity of a solution; numerically

equal to 7 for neutral solutions, increasing with increasing alkalinity and decreasing with

increasing acidity.

Temperature: An indicator of the thermal energy contained in a solid or fluid. Units are

degrees Centigrade (°C) or degrees Fahrenheit (°F). Temperature measurements are made with a

mercury-filled thermometer, bimetallic-element thermometer, or electrical thermistor.

OU3 SOP 10

Rev. No. 0

Date: September 26, 2007

Page 2 of 7

Turbidity: a measure of cloudiness in water due to suspended and colloidal organic and

inorganic material. Turbidity is measured by using a field portable nephlometer capable of

reading down to 0.1 Nephelometric Turbidity Units (NTU).

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated

with them. This list is not intended to be comprehensive and often, additional personnel may be

involved. Project team member information will be included in project-specific plans (e.g., work

plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult

the appropriate documents to determine project-specific roles and responsibilities. In addition,

one person may serve in more than one role on any given project.

Project Manager: Responsible for identifying the appropriate equipment necessary for adequate

site characterization and the requirements for the project-specific tasks.

Quality Control Manager: Performs field program audits and ensures project data quality

objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements

the field program, and supervises other field staff to ensure proper calibration and use of field

equipment through the duration of the project.

Field Technician (or other designated personnel): Responsible for providing requested

instrumentation and basic instructions on its calibration and use. Assists the FTL with the

implementation of tasks and is responsible for regular equipment maintenance and calibration.

4.0 GUIDELINES

This SOP provides a summary of the calibration and operating procedures in accordance with the

various manufacturers' instruction manuals, which accompany each piece of equipment. This

OU3 SOP 10

Rev. No. 0

Date: September 26, 2007

Page 3 of 7

SOP will be reviewed and used in conjunction with the manufacturer's instruction manual by

field team members when using field equipment.

4.1 Applicability

Field equipment must be kept in designated cases, packaged properly, and secured during

transport. This will prevent equipment damage, which may result in inaccurate readings. All

equipment will be decontaminated and calibrated prior to use. As part of the calibration process,

standard laboratory procedures of decontamination are to be followed; prior to calibration and

between calibration buffers, solution vessels and probes are to be rinsed a minimum of three

times with distilled/deionized water and a minimum of one time with the calibration buffer

solution or sample solution.

The meters will always be calibrated according to the manufacturer's instructions before the start

of each workday and whenever equipment drift is suspected. Consult the specific instruments'

instruction manual for further calibration details. The manufacturer's instruction on operation

and maintenance will be follwed.

4.2 pH Meters

Determining pH is critical for predicting and interpreting the reactions and migration of dissolved

chemical constituents in groundwater or surface water. Whenever groundwater or surface water

samples are collected, pH may be measured using a flow-through type meter or another type of

pH probe. Meters used will have temperature and slope adjustments and a repeatability of  $\pm 0.01$ 

standard pH units.

Calibration: Calibration standard buffers will not be used beyond the date specified by the

manufacturer and calibration standard buffers will be stored in a manner that protects the

integrity and precision of the solution. Prior to calibrating the instrument, proper

OU3 SOP 10

decontamination of the equipment will be performed following standard lab practices (refer to

section 4.1).

Two pH standard buffers will be used for calibration of the electrode and are to bracket the

anticipated pH of the groundwater or surface water samples. For example, if the anticipated pH

of sample water is 6, calibration will be conducted with pH 4 and pH 7 buffer solutions; for an

anticipated pH of 8, calibrate with pH 7 and pH 10 buffers. Buffers and samples will be kept at

similar temperature. This action will eliminate the need to correct values for temperature effects.

Theoretically, buffer solutions are stable indefinitely; however, they are susceptible to

contamination. Therefore, old, partially full bottles will be replaced and solutions will not be

used past the manufacturer's recommended expiration date. The instrument calibration will be

checked periodically against a standard solution.

4.3 Specific Conductance Meters

Specific conductance is used as an indicator of water quality. It is a simple indicator of the

change within a system and provides useful information for site characterization. Any meter

used to collect field specific conductance measurements will be equipped with a temperature

compensator, and read directly in micromhos per centimeter (µmhos/cm) corrected to 25°C. The

meter will be calibrated to record values over the anticipated range of conductivity values during

measurement.

Calibration: Reagent-grade potassium chloride (KCI) will be used for the calibration of specific

conductance equipment. Calibration standards will not be used beyond the date specified by the

manufacturer. Consult the manufacturer's instruction manual for further details. Specific

conductance readings will be reported on the field logs in micromhos/centimeter (µm/cm) or

millimhos/cm. The instrument calibration will be checked periodically against a standard

solution of KCl.

OU3 SOP 10

Rev. No. 0

Date: September 26, 2007

Page 5 of 7

4.4 Turbidity Meters

Turbidity meters measure the amount of light scattered at right angles from a beam of light

passing through the test sample. Turbidity readings are the measure of the interaction of light

with suspended solid particles in the sample. Test results are read directly in Nephelometric

Turbidity Units (NTUs) on an LCD digital readout.

Calibration: The turbidity meter is pre-calibrated in the factory, and a simple standardization is

the only step required prior to testing.

4.5 Dissolved Oxygen Meters

Dissolved oxygen (DO) meters measure the quantity of oxygen dissolved in

water. In a typical DO meter, the tip of the probe consists of a cell enclosed by a selective

membrane in a protective holder containing the electrolyte and electrodes.

Calibration: Always calibrate the instrument according to the manufacturer's specifications. For

an accurate calibration, the probe may require immersion in water in an airtight container. If an

open container is used for calibration, the margin of error is approximately 0.1 ppm. If the

calibration is performed above sea level, a correction will be made for the difference in altitude.

Certain table listings are available for oxygen solubility as a function of temperature and salinity.

Refer to the manufacturer's specifications regarding slope calibration.

5.0 QUALITY ASSURANCE AND QUALITY CONTROL

All equipment calibration data must be documented in the field logbooks and/or field forms,

including rationales deviations from this SOP or manufacturer's recommendations. The Field

Team Leader or designated QA reviewer will check and verify that field documentation has been

completed per this procedure and other procedures referenced herein. All equipment must be

operated according to the manufacturer's specifications, including calibration and maintenance.

OU3 SOP 10

6.0 DECONTAMINATION

All equipment used in the sampling process shall be decontaminated prior to field use and

between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall

don appropriate personal protective equipment as specified in the health and safety plan. Any

investigation-derived waste generated in the calibration process shall be managed in accordance

with the procedures outlined in SOP-12.

7.0 REFERENCES

Beta Technology Incorporated. Hydac Instruction Manual for the Hydac Conductivity Temperature pH Tester, Beta Technology Incorporated, 545 Moose Lodge Road, Cambridge,

MD 21613-0265.

Orion, 1984. Research, Incorporated, Laboratory Products Group, Model SA 250 pH Meter

Instruction Manual, 840 Memorial Drive, Cambridge, MA 02139.

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PO Box 329, Chestertown, Maryland, 21620.

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Date: September 26, 2007

OU3 SOP 11 (Rev. 0)

Title: GPS (Global Positioning System) DATA COLLECTION

APPROVALS:

TEAM MEMBER

**BPA Remedial Project Manager** 

**SOP Author** 

SIGNATURE/TITLE,

DATE

9/26/07

9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	

OU3 SOP 11 Rev. No. 0 Date: September 26, 2007 Page 1 of 5

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is a general guidance document for the collection of

coordinates of point locations using a Global Positioning System (GPS) unit. The GPS is a

worldwide, satellite-based system with location positioning capabilities. The system is

administered and managed by the Department of Defense. It is comprised of:

a space segment of approximately 24 operational satellites in complimentary orbit,

a ground control segment made up of a network of control stations around the globe, and

a user segment, which includes anyone who uses GPS to collect locational information.

The system utilizes precise time and radio signals to determine distances from satellites to user

GPS receivers. Distances are most commonly calculated by using the time it takes for a radio

signal code to be transmitted from the satellite and received by the GPS unit. Precise time is

critical to the successful operation of the system. The control stations ensure that the satellites

employ synchronized, atomic clock-derived universal time coordinates (UTC), commonly known

as Greenwich Mean Time (GMT). Receiver units collecting four satellite signals can determine

the geodetic (x, y, z) location through a process of mathematical triangulation. The satellite

signals contain precise time and satellite position information.

GPS technology is used as a method of accurately determining the coordinates of point locations.

The three-dimensional position, or the x, y, and z geodetic coordinates, are determined for the

point locations; however, only the x and y values are primarily used. This is due to the processes

involved in the system; the vertical GPS coordinates are approximately half as accurate as the

horizontal GPS coordinates. The position reported by the GPS unit is based on the geodetic

model selected. The vertical, or z coordinate, value is not as accurate as the reported position due

to the geometry of the satellite constellation relative to the receivers position on the earth.

GPS is one of the arrays of tools for accurately determining location in the field. The collection

of x, y, and z coordinates (for gross data collection) for locations in the field using GPS is useful

OU3 SOP 11

Rev. No. 0

Date: September 26, 2007

for a variety of purposes, including accurate sample locations, locational correlation of remotely

sensed data with ground truth locations, and efficiently collecting better spatial data.

2.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated

with them. This list is not intended to be comprehensive and often, additional personnel may be

involved. Project team member information will be included in project-specific plans (e.g., work

plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult

the appropriate documents to determine project-specific roles and responsibilities. In addition,

one person may serve in more than one role on any given project.

Project Manager: Selects project-specific field documentation with input from other key

project staff, and appropriate oversite agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and

quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods,

performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements

the sampling program, supervises other sampling personnel, and ensures compliance with SOPs

and QA/QC requirements. Prepares daily logs of field activities.

Field Technician (or other designated personnel): Assists the FTL and/or field geologist,

hydrogeologist, or engineer in the implementation of field tasks and field documentation.

3.0 PROCEDURES

There has been, and will continue to be, a considerable and rapid evolution in GPS techniques

and technologies. The procedures described here are to be general in nature and geared towards

hand-held recreational GPS units. Consult the specific instruments' instruction manual for

details on operation.

OU3 SOP 11

Rev. No. 0

Date: September 26, 2007

Page 3 of 5

#### Method

- Turn GPS unit on by pressing and releasing the power key.
- Following the Welcome Page the Satellite Page will be next.
- After sufficient satellites have been acquired, change to Setup
- Set GPS to report coordinates in UTM Zone 11 North, meters, with WGS84 datum
- Change to Position Page.
- When locating a sample location, use the sample identification as described in the field sampling plan
- To record a location press the Mark key; the X Coordinate (Easting), Y Coordinate (Northing), time, and date will be saved. Record the information into a field log book and field data sheets, and save the information in the GPS unit with a unique identification name and/or number to be downloaded later. Coordinates, in meters, should be recorded with 2 decimal places.
- Then enter OK to return to the Position Page.
- To turn off the GPS unit press and hold down the power key.

# 4.0 QUALITY ASSURANCE AND QUALITY CONTROL

Only data with high geometrical strength or low percent dilution of position (PDOP) will be used to ensure high accuracy. The Field Team Leader or designated QA reviewer will check and verify that the GPS coordinates are collected using the appropriate Datum, are entered into a field logbook or electronic database on a daily basis and that coordinates entered into project records match those recorded in the GPS-unit memory. If any corrections are necessary, the field team

OU3 SOP 11

lead or other field personnel will make those corrections before coordinates are transmitted to data users.

All GPS equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

#### 5.0 REFERENCES

U.S Environmental Protection Office of Environmental Information. Global Positioning Systems-Technical Implementation Guidence. September 2003. Available online at <a href="http://www.epa.gov/OEI/pdf/GPS-TIG.pdf">http://www.epa.gov/OEI/pdf/GPS-TIG.pdf</a>

Garmin GPSmap 60CSx Operators Manual.

OU3 SOP 11 Rev. No. 0

Date: September 26, 2007

Page 5 of 5

Date: September 26, 2007

OU3 SOP 12 (Rev. 0)

Title: INVESTIGATION DERIVED WASTE (IDW) MANAGEMENT

APPROVALS:

TEAM MEMBER

EPA Remedial Project Manager

**SOP Author** 

SIGNATURE/TITLE

Revision Number		Reaso	n for Revision		
0	09/26/200			•	٠.
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**OU3 SOP 12** Rev. No. 0

Date: September 26, 2007 Page 1 of 25

#### 1.0 INTRODUCTION

This SOP is based on MWH SOP-07, Investigation-Derived Waste (IDW) Management, Revision 1.0, April 2007, modified for use at the Libby Asbestos Superfund Site OU3. IDW may be generated during field investigations at the Libby Asbestos Superfund Site OU3. The National Contingency Plan (NCP), codified in 40 Code of Federal Regulations (CFR) 300, requires that IDW be handled to attain all the applicable or relevant and appropriate requirements (ARARs) to the extent practicable, considering the urgency of the situation. The purpose of this SOP is to present procedures to be followed in the management of IDW generated during the field investigations.

Typical IDW generated during field activities are solid wastes and may include (but are not limited to) the following media and waste types:

Fluids	Solids	
Purge water and groundwater	Soils and soil cuttings	
Drilling mud	Plastic tarps or sheeting	
Grout	Drill pipe and well casing/screen	
Decontamination fluids and wastewater	Decontamination solids	
	Disposable equipment (i.e., rope, bailers, sampling equipment, & other consumables)	
	Spent personal protective equipment (PPE)	
	Used containers, sample bottles	
	Packaging materials	

The above wastes may or may not be encountered, generated or managed while performing field investigations. However, all solid waste streams will be characterized to determine if they are hazardous wastes per 40 CFR § 262.11 for the purposes of handling and disposal. Guidance from this document shall be used as part of project planning to estimate total volumes of IDW likely to be generated as well as how the IDW will be managed and disposed.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in IDW handling must follow health and safety protocols described in the

health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen

by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the

lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung

cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases

(ATSDR 2006).

3.0 DEFINITIONS

Area of Contamination (AOC) unit: The AOC unit concept is critical to the IDW management

at a CERCLA investigation site. Although EPA has not promulgated a definition of an AOC

unit, an AOC unit is generally an area within a CERCLA investigation site with similar

characteristics with respect to contamination and the associated risks to human health and the

environment. A CERCLA investigation site may contain one or more AOC units.

**Decontamination fluids:** Any fluids, including aqueous wash water, solvents, and contaminants

that are used or generated during decontamination procedures.

Decontamination solids: Any solids, including soils and soil cuttings, fill materials, and

contaminants that are generated during decontamination procedures.

Grout: A fluid mixture of cement and water (neat cement) of a consistency that can be forced

through a pipe and placed as required.

Hazardous waste: A solid waste that meets the definition of a hazardous waste under RCRA as

defined in 40 CFR § 261.3.

Investigation-derived waste (IDW): Solid wastes, as defined in 40 CFR § 261.2, directly

generated as result of performing the field activities.

OU3 SOP 12

Rev. No. 0

Date: September 26, 2007

Page 3 of 25

Nonhazardous waste: A solid waste that does not meet the definition of a hazardous waste as

defined in 40 CFR § 261.3 or is excluded from hazardous waste regulation per 40 CFR §

261.4(b).

Soils and soil cuttings: Solid material generated from excavation or drilling processes. Soils

may include native soils, fill materials, and/or other historical plant waste streams used as fill

materials on the site.

Solid waste: Any waste stream (solid, liquid or containerized gas) that meets the definition of

solid waste under RCRA as defined in 40 CFR § 261.2.

4.0 RESPONSIBILITIES

This section presents a brief definition of the field team roles and responsibilities for

management of IDW generated while conducting field investigations. This list is not intended to

be a comprehensive list as additional personnel may be involved. Project team member

information shall be included in project-specific plans (e.g., work plan, field sampling plan

(FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate

documents to determine project-specific roles and responsibilities. In addition, one person may

serve in more than one role on any given project.

Project Manager: Responsible to ensure that all field team members are properly trained per

their responsibilities associated with IDW and that appropriate equipment and facilities are

available for appropriate IDW management.

Field Team Leader (FTL): Implements the field program and supervises all field team

members in the appropriate management of IDW. Ensures that only properly trained personnel

are managing IDW on the site.

Environmental, Health and Safety (EHS) Officer: Assists the Team Leader in the supervision

of all IDW management on site. The EHS officer shall be responsible for all IDW identification

and characterization, on site disposal, off site shipment and disposal, waste accumulation,

emergency response and contingency planning, IDW training, and IDW reporting and

recordkeeping.

Project Team Members: Ensure that they are properly trained prior to any IDW management as

well as follow the appropriate IDW procedures and training.

5.0 REGULATORY BASIS AND GUIDANCE

IDW encountered, generated, or managed during the field investigations may contain hazardous

substances as defined by CERCLA. Some IDW may be hazardous wastes under RCRA while

others may be regulated under other federal laws such as TSCA. These regulatory requirements

may be applicable or relevant and appropriate requirements (ARARs) which impact how the

IDW is managed.

5.1 EPA Guidance on IDW Management

The management of IDW shall be in accordance with EPA Guidance "Management of

Investigation-Derived Wastes During Site Inspections", May 1991 (EPA, 1991). The specific

elements of EPA's guidance for IDW management are as follows:

• Characterizing IDW through the use of existing information (manifests, MSDSs,

previous test results, knowledge of the waste generation process, and other relevant

records) and best professional judgement.

Delineating an AOC unit for leaving RCRA hazardous soil cuttings within the unit.

OU3 SOP 12

Rev. No. 0 Date: September 26, 2007

Page 5 of 25

• Containerizing and disposing of RCRA hazardous groundwater, decontamination

fluids, PPE, and disposable equipment at RCRA Subtitle C facilities.

• Leaving on-site RCRA nonhazardous soil cuttings, groundwater, and decontamination

fluids preferably without containerization and testing.

In general, EPA does not recommend removal of wastes from sites, in particular, from those sites

where IDW do not pose any immediate threat to human health or the environment. Actions taken

during field investigations with respect to IDW that leave conditions essentially unchanged

should not require a detailed analysis of ARARs or assurance that conditions at the site after field

investigations will comply with the ARARs. At the same time, field personnel ensure that their

handling of IDW does not create additional hazards at the site.

In brief, compliance with the NCP can generally be assured by:

1) Identifying contaminants, if any, present in the IDW based upon existing information and

best professional judgement; testing is not required in most circumstances.

2) Determining ARARs and the extent to which it is practicable to comply with them.

3) Delineating an AOC unit based upon existing information and visual observation if soil

cuttings are RCRA hazardous.

4) Burying RCRA hazardous soil cuttings within the AOC unit, so long as no increased hazard

to human health and the environment will be created. Containerization and testing are not

required.

5) Containerizing RCRA hazardous groundwater and other RCRA hazardous IDW such as

PPE, disposable sampling equipment, and decontamination fluids for off-site disposal.

5.2 Hazardous Waste Regulation

The RCRA hazardous waste regulations are clearly ARARs for hazardous IDW generated and

managed during field investigations. However, with the application of EPA IDW guidance,

RCRA requirements apply to management of IDW in the following manner:

• If RCRA hazardous IDW is stored or disposed off-site, then comply with all RCRA

(and other ARAR) requirements.

• If RCRA hazardous IDW is stored on-site, then comply with RCRA (and other ARAR)

requirements to the extent practicable.

For these field investigations, the following general guidance is expected to be practicable and

therefore followed, recognizing that each situation will be evaluated against EPA IDW guidance

(EPA, 1991) as well as RCRA hazardous waste requirements and other ARARs:

• IDW may be assumed not to be a "listed" hazardous waste under RCRA 40 CFR 261

Subpart D, unless available information about the site suggests otherwise.

• IDW characterization to determine if the IDW exhibits RCRA hazardous waste

characteristics do not typically require testing if the characterization can be made by

"applying knowledge of the hazardous characteristics in light of the materials or

processes used" or by historical testing consistent with 40 CFR § 262.11(c).

Compliance with the RCRA hazardous waste generator requirements of 40 CFR Part

262 for all RCRA hazardous IDW generated and/or managed (with exception of soil

cuttings managed in accordance with the EPA IDW guidance). It is presumed that the

RCRA hazardous IDW generated will fall within the large quantity generator (LOG)

requirements.

OU3 SOP 12

Rev. No. 0

Date: September 26, 2007

Page 7 of 25

- Land disposal does <u>not</u> occur (and thus the Land Disposal Restrictions [LDR] of 40
   CFR Part 268 are <u>not</u> applicable) when IDW soil cutting wastes are:
  - Moved, stored or left in place within a single AOC unit;
  - Capped in place;
  - Treated in situ (without moving the IDW to another AOC unit for treatment);
     or
  - Processed within the AOC unit to improve structural stability (without placing the IDW into another AOC unit for processing).
- Conversely, land disposal <u>does</u> occur (and the LDR of 40 CFR Part 268 <u>are</u> applicable)
   when IDW soil cutting wastes are:
  - Moved from one AOC unit to another AOC unit for disposal;
  - Moved outside an AOC unit for treatment or storage and returned to the same AOC unit for disposal;
  - Excavated from an AOC unit and placed in a container, tank, surface impoundment, etc. and then re-deposited back into the same AOC.

#### 5.3 TSCA PCB Regulation

IDW containing PCBs at detectable levels may be generated, although the concentration of PCBs in any IDW generated is expected to be far below 50 ppm. However, IDW generated will be evaluated for PCBs and managed according to the following per the requirements of 40 CFR Part 761 Subpart D:

- Liquid IDW at concentrations greater than or equal to 50 ppm PCBs will be incinerated off-site at a TSCA-approved incinerator site.
- Nonliquid IDW at concentration greater than or equal to 50 ppm PCBs may be incinerated, treated by an equivalent TSCA-approved method, or disposed in a TSCA chemical landfill off-site.

OU3 SOP 12

Rev. No. 0

Date: September 26, 2007

Page 8 of 25

IDW at concentrations less than 50 ppm are generally not regulated under TSCA, and

may be disposed in an acceptable Subtitle D facility.

6.0 DESCRIPTION OF ANTICIPATED IDW MANAGEMENT

The following subsections provide a description of the anticipated IDW to be encountered,

generated, and/or managed at the Libby Asbestos Superfund Site OU3 during field activities and

the anticipated management of each. It should be noted that this information is provided for

planning purposes, and will be evaluated and may need to be revised based upon actual

experience while on site.

6.1 Soil and Soil Cuttings

During field investigations, surface soil samples, samples of mine waste rock, and samples of

fine tailings will be collected. Only a small portion of material will be collected for analysis.

While the soil and soil cuttings IDW generated will be evaluated on a case-by-case basis, the

general approach will follow the EPA guidance for IDW (EPA, 1991) which includes:

• Characterizing the IDW through the use of existing information (previous test results,

previous waste characterization, knowledge of the waste generation process, and other

relevant records) and best professional judgement.

• Soil and soil cuttings which are not used directly for sample makeup will not be taken

outside of the AOC unit in which they were generated.

Soil and soil cuttings within the AOC where they are generated will be placed back into

the same investigation pit, trench, or bore hole and in the same order from which the

material was removed, to the extent practicable and unless noted otherwise in the FSP.

OU3 SOP 12

Rev. No. 0.

Date: September 26, 2007

Page 9 of 25

• Soil cuttings potentially requiring RCRA disposal will be handled per the procedures

presented in Section 7.0 below and disposed in an off-site RCRA facility.

6.2 Spent Sampling-Related Equipment

During field investigations, spent sampling-related equipment will be generated. This may

include (but not limited to) plastic sheeting/tarps, rope, bailers, sampling equipment, spent PPE,

sample bottles, used containers, packaging materials, and other consumables. Although the vast

majority of the spent sampling-related equipment is expected to be nonhazardous, these IDW

may contain a listed hazardous waste (e.g., spent solvents) or may exhibit a hazardous waste

characteristic (e.g., toxicity from metals).

While the spent sampling-related equipment will be evaluated on a case-by-case basis, the

general approach to be followed for spent sampling-related equipment IDW will follow the EPA

guidance for IDW (EPA, 1991) which includes:

Containerizing the spent sampling-related equipment, typically in a satellite

accumulation station.

• Characterizing the spent sampling-related equipment IDW through the use of existing

information (previous test results, previous waste characterization, knowledge of the

contaminants present, and other relevant records) and best professional judgement.

This characterization will be documented and maintained as part of the solid/hazardous

waste determination records.

• Those spent sampling-related equipment IDW that are determined to be nonhazardous

will be disposed of onsite or as municipal waste.

• Those spent sampling-related equipment IDW that are determined to be hazardous will

be managed per the procedures presented in Section 7.0 below and disposed in an off-

site RCRA facility.

6.3 Decontamination Fluids and Solids

During field investigations, decontamination fluids and solids will be generated. Typically, these

will be generated at a common decon area, although there may be more than one decon area.

Typically, the decontamination IDW will include (but not limited to) washwater from

vehicles/equipment, and cleaning agents. Although the vast majority of decontamination IDW is

expected to be nonhazardous, this IDW may contain a listed hazardous waste (e.g., spent

solvents) or may exhibit a hazardous waste characteristic (e.g., toxicity from metals).

While the decontamination IDW will be evaluated on a case-by-case basis, the general approach

to be followed for decontamination IDW will follow the EPA guidance for IDW (EPA, 1991)

which includes:

• Containment of decontamination fluids (typically washwater) as generated. The

washwater will be segregated from solids to the extent practicable (i.e., solids will be

allowed to settle out of the washwater on the decontamination containment pad).

Washwater will then be containerized to await waste determination. Solids will also be

containerized in a separate container to await waste determination.

• Other decontamination solids such as cleaning utensils and PPE will also be

containerized to await waste determination.

• Characterizing the decontamination IDW through the use of existing information

(previous test results, previous waste characterization, knowledge of the contaminants

present, and other relevant records) and best professional judgement. This

OU3 SOP 12

characterization will be documented and maintained as part of the solid/hazardous

waste determination records.

• The decontamination solids IDW that are determined to be nonhazardous will be

disposed of onsite.

• The decontamination liquids IDW that are determined to be nonhazardous will be

disposed as a nonhazardous solid waste, preferably on-site.

• The decontamination IDW (either liquid or solid) that are determined to be hazardous

will be managed per the procedures presented in Section 7.0 below and disposed in an

off-site RCRA facility.

6.4 Drilling, Well Purging, and Development Water

Generally, water at the Site that is extracted from boreholes, wells or piezometers for the purpose

of drilling, development, sampling, or hydraulic testing is considered non-hazardous and will be

discharged to designated shallow sumps away from the boreholes or wells at the site. If the water

generated is determined to be hazardous will be managed per the procedures presented in Section

7.0 below and disposed in an off-site RCRA facility.

7.0 PROCEDURES FOR HAZARDOUS IDW MANAGEMENT

The following procedures apply to all IDW that have been determined to be hazardous except for

soil cuttings IDW that remain with the AOC unit.

7.1 Introduction

Once an IDW has been determined to be hazardous, the federal RCRA Subtitle C waste

management requirements apply to that waste. The scope of this procedure covers the

OU3 SOP 12

Rev. No. 0

Date: September 26, 2007

Page 12 of 25

requirements for large quantity generators (LQG) of hazardous IDW which manage the

hazardous IDW on site such that RCRA permitting is not required.

7.2 Determine Land Disposal Restrictions

The 1984 amendments to the RCRA law included a prohibition of land disposal of certain

hazardous wastes without first meeting some treatment standards. For the most part, all listed

and characteristic hazardous wastes must be treated according to the treatment levels and

technologies outlined in 40 CFR Part 268 to reduce the toxicity and/or mobility of hazardous

constituents prior to being disposed of on the land, i.e., landfilled. Therefore, a generator must

determine if the waste is a "restricted waste" under the land ban rules, and if so, off site treatment

and disposal is limited. Note that these rules apply only to wastes destined for land disposal

which is defined as: placement in or on the land including a landfill, surface impoundment,

waste pile, injection well, land treatment facility, salt dome formation, salt bed formation,

underground mine or cave, or concrete vault or bunker. Wastes which are shipped off site for

disposal other than land disposal are not regulated under the land disposal restriction regulations

of 40 CFR Part 268.

Generators of hazardous wastes must determine if the waste is restricted from land disposal

under 40 CFR Part 268. The following reporting and recordkeeping requirements apply.

• If a generator determines that he is managing a restricted waste and the waste does not

meet the applicable treatment standards, with each shipment of waste, the generator

must notify the treatment or storage facility in writing of the appropriate treatment

standards:

If the generator determines that he is managing a restricted waste and the waste can be

disposed without further treatment, with each shipment of waste, the generator must

submit to the treatment, storage or disposal facility a notice and certification stating

that the waste meets the applicable treatment standards;

OU3 SOP 12

Rev. No. 0

Date: September 26, 2007

Page 13 of 25

• If the generator determines that he is managing a waste subject to an exemption from a prohibition on the type of land disposal method utilized for the waste, with each shipment of waste, the generator must submit to the receiving facility a notice stating that the waste is not prohibited from land disposal;

• If the generator is managing prohibited waste in tanks, containers, or containment buildings regulated under 40 CFR 262.34, and is treating such waste in such tanks, containers, or containment buildings to meet applicable treatment standards, the generator must develop a waste analysis plan which describes the procedures the generator will carry out to comply with the treatment standards; and

 If the generator determines whether the waste is restricted based solely on his knowledge of the waste, all supporting data used to make this determination must be retained on-site in the generator's files.

The generator must retain on-site a copy of all notices, certifications, demonstrations, waste analysis data, and other documentation produced pursuant to these requirements for at least three years from the date the waste was last shipped from the site. It should also be noted that it is prohibited to dilute a hazardous waste in order to circumvent the land disposal prohibitions (40 CFR 268.3). Once a waste is determined to be a "restricted waste", an appropriate Treatment, Storage, and Disposal Facility (TSDF) can be selected to properly treat and dispose of the waste.

#### 7.3 On-Site Accumulation

As discussed in Section 5.0 above for each IDW generated, a large quantity generator (LQG) must make the appropriate hazardous waste determination per 40 CFR Part 262.11. If the IDW is determined to be hazardous, then the IDW will typically be stored on-site prior to shipment off-site for disposal. The following requirements apply to all hazardous IDW being stored on-site prior to shipment.

7.3.1 EPA Identification Number (40 CFR Part 262.12)

Any facility which is a LQG of hazardous wastes must not treat, store, dispose, transport or offer

for transportation any hazardous waste without first obtaining a EPA identification number from

EPA (or the authorized state). Hazardous wastes cannot be offered to transporters or to

treatment, storage or disposal facilities that have not received a EPA identification number.

7.3.2 On-Site Hazardous Waste Accumulation (Storage) (40 CFR 262.34(d))

Two types of accumulation areas for hazardous waste are permissible for a LQG without RCRA

interim status or a Part B permit. These are the "90-day storage area" and the "satellite

accumulation station" (SAS). The SAS requirements are discussed below. With regards to a

"90-day storage area", a LQG may store hazardous wastes on-site for up to 90 days or less in a

storage area, provided that the following conditions are met:

• If the waste is placed in containers, the requirements of 40 CFR Part 265 Subpart I

(container requirements) are met. See below for container requirements;

• If the waste is placed in tanks, the requirements of 40 CFR 265 Subpart J (tank

requirements) are met. See below for the tank requirements.

• At closure, the generator closes the storage area per the requirements of 40 CFR

265.111 and 40 CFR 265.114;

• The date which the hazardous waste is placed in the storage area is clearly marked on

the container, and the container is clearly marked as "Hazardous Waste";

• The facility complies with 40 CFR Part 265 Subpart C, Preparedness and Prevention

(See Section 6.3.3 below);

• The facility complies with 40 CFR Part 265 Subpart D, Contingency Plan and

Emergency Procedures (See Section 6.3.4);

The facility complies with 40 CFR Part 265.16 training requirements (See Section 6.6

below);

OU3 SOP 12

Rev. No. 0

Date: September 26, 2007

Page 15 of 25

 Any hazardous wastes which are stored longer than 90 days must first be granted an extension by EPA (or authorized state).

### 90-Day Storage Area Container Requirements (40 CFR Part 265 Subpart I)

Hazardous waste stored in containers must meet the following requirements:

- Containers must be in good condition, free of leaks;
- Hazardous wastes must be compatible with container (or liner) material;
- Containers must always be kept closed except to add or remove wastes;
- Containers must be handled in a manner to avoid ruptures;
- The storage area must be inspected at least weekly to check for container deterioration; and
- Incompatible wastes must be stored separately with separate secondary containment.

Incompatible wastes are wastes that are unsuitable for co-mingling because the co-mingling could result in any of the following:

- Extreme heat or pressure generation;
- Fire:
- Explosion or violent reaction;
- Formation of substances that have the potential to react violently;
- Formation of toxic dusts, mists, fumes, gases, or other chemicals; and/or
- Volatization of ignitable or toxic chemicals due to heat generation.

90-Day Storage Area Tank Requirements (40 CFR Subpart J)

LQGs that accumulate or store hazardous wastes in tanks or tank systems must meet the

following requirements:

• For tanks existing prior to July 14, 1986, an assessment of tank must be performed and

certified by an independent, qualified, licensed engineer. The written certification

must be kept on file at the facility (40 CFR 265.191);

• New tank systems (those built after July 14, 1986) must meet tank technical standards

and have been certified by an independent, qualified, licensed engineer. The written

certification must be kept on file at the facility (40 CFR 265.192);

• New tank systems must have adequate secondary containment and leak detection

systems. Existing tanks must be upgraded to meet these standards by the time the

tank is 15 years of age (40 CFR 265.193);

Tanks must be operated to prevent system failure, overflow and spills. Tanks must be

operated with sufficient freeboard to prevent overtopping (40 CFR 265.194);

• Inspect the tanks at least once each operating day for the following:

Discharge control equipment;

Monitoring equipment and controls;

Tank level; and

- Evidence of leaks or spills. (40 CFR 265.195)

• Inspect the tanks at least weekly for corrosion, erosion or leaks;

• The tank must meet the closure and post-closure care provisions of 40 CFR

265.197; and

Store incompatible wastes separately (40 CFR 265.199).

OU3 SOP 12 Rev. No. 0

Date: September 26, 2007

Page 17 of 25

Satellite Accumulation Station (SAS) Requirements (40 CFR 262.34(c))

A SAS is a container placed at or near the point of waste generation for the purpose of collecting

the waste as it is being generated. For example, a container may be placed in the quality control

laboratory for collection of hazardous wastes generated in the laboratory. This SAS may collect

up to 55 gallons of hazardous waste or 1 quart of acute hazardous waste. The SAS does not need

to meet the requirements of a storage area, provided the following conditions are met:

• The amount of hazardous waste accumulated at the SAS does not exceed 55 gallons

(or 1 quart of acute hazardous waste);

• The SAS is located at or near the point of generation where the waste is initially

accumulated and is under the control of the operator of the process generating the

waste;

• The container used is in good condition, is compatible with the wastes being

accumulated, and is kept closed except to add or remove wastes;

The container is marked with the words "Hazardous Waste" or other words to identify

the contents; and

Once the 55-gallon limit is reached, the date is marked on the container and the

container is moved from the SAS within three days to a proper location. For

example, the wastes must either be moved to the storage area or be picked up by a

waste transporter and moved off-site.

7.3.3 Preparedness and Prevention (40 CFR Part 265 Subpart C)

The following preparedness and prevention steps must be taken concerning the hazardous waste

storage area:

• The storage area must be operated and maintained to minimize the possibility of fire,

explosions or releases of hazardous waste;

The facility must have appropriate communication systems, fire-fighting equipment,

spill control equipment and decontamination equipment;

OU3 SOP 12 Rev. No. 0

Date: September 26, 2007

Page 18 of 25

• All emergency response systems and equipment must be tested monthly with

documentation and maintained to assure proper operation;

Persons handling hazardous wastes must have immediate access to alarms and/or

communication systems;

The storage area shall have adequate aisle space for emergency response activities;

and

• The facility must attempt to make arrangements with the local police, fire

departments, emergency response teams, and local hospitals to assure readiness for

potential emergencies associated with the storage area.

7.3.4 Contingency Plan and Emergency Procedures (40 CFR Subpart D)

A LQG that accumulates or stores hazardous waste on site in a 90-day storage area must develop

and keep current a contingency plan for the facility. The purpose of the contingency plan is to

provide an organized plan of action and delegation of responsibilities and authority to specific

facility personnel to respond to emergency situations that may require both the facility and/or

outside resources. The contingency plan is designed to minimize hazards to humans or the

environment from fires, explosion or any unplanned sudden or non-sudden release of hazardous

waste/hazardous waste constituent to air, soil or surface water in compliance with the

requirements of 40 CFR 265 Subpart D. A Contingency Plan will be maintained on the site if

hazardous IDW are accumulated on-site.

The key components of the contingency plan include the following (40 CFR 265.52):

• A description of the emergency response organization, including designation of the

Emergency Coordinator and alternates;

Response procedures;

Emergency notification;

OU3 SOP 12

Rev. No. 0

Date: September 26, 2007

• Arrangements with local authorities;

• List of names, addresses and phone numbers of designated emergency personnel and

alternates;

List of emergency response communication equipment and locations;

• Evacuation procedures, routes and alternates; and

Procedures for amending the plan.

Copies of the plan must be sent to (40 CFR 265.53):

The Project Manager;

Lincoln County Sheriff's department;

• Libby fire department; and

• Other agencies as deemed appropriate.

The emergency coordinator (EC) is the key person facilitating emergency preparedness and

response. The EC or designated alternate shall be on-site or on-call at all times. The EC and

alternates must be trained and thoroughly familiar with the contingency plan, emergency

response activities and operation of the facility. The EC must know the locations and

characteristics of all waste generated, location of all records within the facility and the facility

layout. The EC must have the authority to commit the resources needed to carry out the spill

response plan. Any person or department who first discovers any spill of a hazardous

waste/material is responsible for notifying the spill response/emergency response coordinator.

The EC for the Libby Mine Site field investigations will be the EHS Officer with the Field Team

Leader and the Project Manager as alternates.

The contingency plan should be reviewed and immediately amended when:

Changes in applicable regulations occur;

• The plan fails in an emergency;

Changes are made to emergency procedures;

• Changes occur in emergency personnel list; or

Changes occur in emergency equipment list.

7.4 Pre-Transportation Requirements

Prior to transporting hazardous wastes or offering hazardous wastes for transportation off-site,

the generator must comply with the following:

• Package the hazardous wastes in DOT-approved containers per 49 CFR Parts 173,

178 and 179. DOT-approved containers (such as drums) are usually marked as being

DOT-approved);

• Label the hazardous wastes according to DOT labeling requirements per 49 CFR Part

172;

Mark each container (of 110 gallons or less) used in transportation with the following:

HAZARDOUS WASTE - Federal Law Prohibits Improper Disposal. If found,

contact the nearest police or public safety authority or the EPA.

Generator's Name and Address

Manifest Document Number

Ensure that the initial transporter placards the transport vehicle with the appropriate

placard in accordance with 49 CFR Part 172 Subpart F.

7.5 Manifesting Off-Site Shipments of Hazardous IDW

Any generator which transports or offers for transportation hazardous waste for off-site

treatment, storage or disposal must prepare a manifest according to manifest instructions for each

shipment of similar hazardous wastes. The manifest must be carefully filled out with each

**OU3 SOP.12** 

Rev. No. 0

Date: September 26, 2007

Page 21 of 25

shipment. Take care to follow the instructions and use the terms as listed in the instructions. A generator must designate on the manifest one facility (designated facility) which is permitted to handle the waste described on the manifest (40 CFR 262.20).

The generator must determine if the state to which the wastes are destined (consignment state) requires use of its own manifest. If so, then the consignment state's manifest must be used. If the consignment state does not require use of its manifest, and the state in which the waste shipment originates (generator state) does, then the manifest from the generator state must be used. If both states have manifests, use the consignment state manifest, making sure that there are sufficient copies to meet the generator state distribution requirements. If neither state requires use of its manifest, then any uniform hazardous waste manifest may be used (40 CFR 262.21).

The manifest must contain at least enough copies such that the generator gets two copies, the transporter gets one copy and the designated facility gets one copy. Some states require additional copies to be sent to the state. At the time of shipment, the generator must keep one copy (the generator copy) of the completed, signed manifest and give the remaining copies to the transporter. Each copy must have the signature of the generator and the transporter at the time of shipment. The original manifest shall be returned to the generator once the shipment reaches the designated facility and the manifest is signed by the designated facility (40 CFR 262.21).

If the original, signed manifest is not received by the generator within a certain number of days, action by the generator is required. These requirements are discussed in the following sections:

- If, after 35 days from the date of shipment, the original manifest copy is not yet received by the LQG, the LQG must contact the transporter and/or the designated disposal facility to determine the status of the hazardous waste (40 CFR 262.42(a)(1)).
- If after 45 days from the date of shipment, the original manifest copy is not yet received by the LQG, the LQG must submit an exception report to the U.S. EPA (or authorized state). The exception report must include a copy of the manifest along

OU3 SOP 12

Rev. No. 0

Date: September 26, 2007

Page 22 of 25

with an explanation of efforts to locate the hazardous wastes and the result of these

efforts (40 CFR 262.42(a)(2)).

7.6 Personnel Training

Any person, and their immediate supervisor(s), involved in waste management at a LQG facility

which stores hazardous waste in a 90-day storage area must undergo initial and annual training

for hazardous waste management (40 CFR 262.34(a)(4) and 40 CFR 265.16). Facility personnel

are required to successfully complete a program of classroom instruction or on-the-job training

that teaches them to perform hazardous waste management duties relevant to their jobs. The

program must be directed by a person trained in hazardous waste management procedures.

The training must be designed to enable personnel to effectively respond to emergencies by

becoming familiar with emergency procedures, emergency equipment and emergency systems,

including the following;

· Procedures for using, inspecting, repairing and replacing facility emergency and

monitoring equipment;

Communications or alarm systems;

Response to fires or explosions; and

• Off-site communication.

Employee training is to be held at regular intervals. Emergency planning information, e.g., the

Contingency Plan, also should be provided to state and local emergency response agencies at

regular intervals (40 CFR 265.37 and 265.53). Employees required to receive the training cannot

work unsupervised until they have completed the training requirements (either classroom or on-

the-iob training). In addition, facility personnel must take part in an annual review of the initial

training.

The following records must be maintained at the facility for employees affected by this training:

**OU3 SOP 12** 

Rev. No. 0

Date: September 26, 2007

Page 23 of 25

• Job title for each position and name of employee filling each job;

Job descriptions for each position related to hazardous waste management;

• Written description of type and amount of initial and continuing training that will be

given to each person filling the various job positions; and

Documentation that necessary training has been given and completed by each affected

personnel.

Training records are required to be kept on current personnel until closure of the facility. For

former employees, training records must be kept for at least three years from the date the

employee last worked at the facility and may be transferred if the employee stays within the same

company (40 CFR 265.16(e).

7.7 Reporting and Recordkeeping

The following reports are required of a LQG:

Manifest exception reports as discussed in Section 6.5 above.

• A LQG must submit a Biennial Report to the EPA (or authorized state) every even

numbered year by March 1, e.g., March 1, 2008 for the 2007 reporting year. The

Biennial Report is to be submitted on EPA form 8700-13A.

The following records are required to be kept for a minimum of three years by the LQG:

The signed original manifests;

• Biennial reports;

Exception reports;

• All records pertaining to hazardous waste determinations; and

OU3 SOP 12

Rev. No. 0

Date: September 26, 2007

Land disposal determination records, notification and certification records.

## 8.0 QUALITY ASSURANCE AND QUALITY CONTROL

All IDW data must be documented in the field logbooks, field forms, manifests, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that IDW documentation has been completed per this procedure and other procedures referenced herein.

### 9.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

EPA, 1991. Management of Investigation-Derived Wastes During Site Inspections, EPA May 1991, EPA/540/G-91/009

OU3 SOP 12 Rev. No. 0 Date: September 26, 2007

Page 25 of 25

Date: September 26, 2007

OU3 SOP 13 (Rev. 0)

Title: GROUNDWATER ELEVATION MEASUREMENTS

APPROVALS:

**TEAM MEMBER** 

EPA Remedial Project Manager

SOP Author

SIGNATURE/TITLE

DATE

9/24/07

9/26/57

Revision Number	Date	Reason for Revision
0	09/26/2007	-
	•	

OU3 SOP 13 Rev. No. 0 Date: September 26, 2007 Page 1 of 9

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is based on MWH SOP-13, Groundwater Elevation

Measurements, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site

OU3. Field measurements of static water levels or depth to groundwater are necessary for the

determination of purge volumes, groundwater elevations, direction of groundwater flow, and

non-aqueous phase liquid (NAPL) thickness. The accuracy, precision, and reliability of these

measurements are dependent on the proper use and care of the field instruments. Consistent

methods and practices of data collection (in accordance with recommended manufacturer

instructions) must be followed to obtain valid and useful data. Common instruments used to

collect groundwater elevation and NAPL thickness data include electric water level indicators

and dual-phase probes, and chalked tape. Due to decontamination difficulties, dual-phase probes

will only be used in wells containing or suspected of containing NAPL. Use of these probes in

"clean" wells may result in cross-contamination. All groundwater elevation measuring

equipment will be decontaminated prior to field use and between wells.

This SOP provides a standard reference for selection of the proper equipment and use of the

proper techniques for measurement of groundwater elevation and NAPL thickness. The purpose

of these procedures is to enable the user to collect representative and defensible groundwater

elevation and NAPL thickness data.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in groundwater elevations measurements must follow health and safety

protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so

small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when

disturbed and when embedded in the lung tissue can cause health problems. Significant exposure

to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung

disease), and other respiratory diseases (ATSDR 2006).

**OU3 SOP 13** 

Rev. No. 0

Date: September 26, 2007

Page 2 of 9

3.0 DEFINITIONS

Dual-Phase Probe: A device consisting of a spool of small-diameter graduated cable or tape

and a weighted probe attached to one end, used to measure NAPL thickness. A buzzer emits an

intermittent signal when the probe contacts water and a steady signal upon contact with NAPL.

Electric Water Level Indicator: A device consisting of a spool of small-diameter graduated

cable or tape and a weighted probe attached to one end, used to measure static water levels. A

buzzer emits a steady signal when the probe contacts water.

Static Water Level: The elevation of the top of a column of water in a monitoring well or

piezometer that is not influenced by pumping or conditions related to well installation,

hydrologic testing, or nearby groundwater extraction.

NAPL: NAPL may be lighter than groundwater (e.g., gasoline), or heavier/denser than

groundwater (e.g., chlorinated solvents). Light non-aqueous phase liquid (LNAPL) is commonly

encountered at the water table surface. Dense non-aqueous phase liquid (DNAPL) is encountered

at the interface between impermeable and permeable materials below the water table.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated

with them. This list is not intended to be comprehensive and often, additional personnel may be

involved. Project team member information will be included in project-specific plans (e.g., work

plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult

the appropriate documents to determine project-specific roles and responsibilities. In addition,

one person may serve in more than one role on any given project.

Project Manager: Selects site-specific measurement methods and locations for groundwater

elevation measurements with input from other key project staff, and applicable oversight

agencies.

OU3 SOP 13

Rev. No. 0

Date: September 26, 2007

Page 3 of 9

Quality Control Manager: Overall management and responsibility for quality assurance and

quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods,

performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements

the sampling program, supervises other sampling personnel, and ensures compliance with SOPs

and QA/QC requirements. Prepares daily logs of field activities.

Field Technician (or other designated personnel): Assists the FTL, project

geologist/hydrogeologist, or engineer in the completion of tasks and is responsible for the proper

use and maintenance of groundwater elevation measuring equipment.

5.0 STATIC GROUNDWATER LEVEL MEASUREMENTS

Introduction

Unless otherwise specified, electric water level indicators will be used to measure depth to water.

Depth to water measurements will be converted to groundwater elevations.

A water-level measurement will be obtained prior to purging the well and also immediately

following sampling. The total depth of the well will also be measured and recorded before

purging the well.

**Measuring Point** 

A measuring point (MP) shall be selected and marked for each monitoring well and

piezometer in which water level measurements will be made. Generally, the MP will be

the top of the well casing on the north side. The MP will be permanently marked using

an indelible marker or a notch cut into the PVC casing. When the top-of-casing elevation

of a monitoring well or piezometer is surveyed, the licensed surveyor shall measure the

OU3 SOP 13

Rev. No. 0

Date: September 26, 2007

Page 4 of 9

MP elevation and reference this measurement to an appropriate datum (such as feet above

mean sea level).

Water Level Measurements

When water levels are measured to describe the groundwater potentiometric surface, the

water level will be measured prior to purging. All wells to be gauged during a monitoring

event and used to construct the potentiometric surface should have water levels taken

within the same 24 hour period.

Water levels will be measured to the nearest 0.01 foot using an electronic water-level

indicator. The measurement will be made to the measuring point reference mark placed

on the top of the PVC well casing.

Prior to use for each measurement, the water level indicator will be decontaminated with

an Alconox® solution followed by a distilled water rinse.

An electronic probe consists of a contact electrode attached to the end of an insulated

electrical cable, and a reel which houses an ammeter, a buzzer, or other closed circuit

indicator. The indicator shows a closed circuit and flow of current when the electrode

touches the water surface. The electronic probes used shall be calibrated periodically by

comparing the depth-to-water readings between the electronic probe and a steel surveyors'

tape.

The procedure for measuring water levels with an electric probe is as follows:

1. Switch on and test that the battery is charged.

2. Lower the probe into the well until the ammeter or buzzer indicates a closed

circuit. Raise and lower the probe slightly until the shortest length of cable that

gives the maximum response on the indicator is found.

OU3 SOP 13

Rev. No. 0

Date: September 26, 2007

Page 5 of 9

3. With the cable in this fixed position, note the depth to water from the Measuring

Point (MP).

4. Repeat as necessary until at least two identical duplicate measurements are

obtained. Record the final measurement with time and day of the measurement.

Calibration of the electronic probe will be checked at regular intervals as part of regular

maintenance measuring the position of the electrode to check that the calibration marks on the

electronic probe correspond to those on the steel surveyors tape.

Well Depth Measurements

The total depth of a well shall be measured by sounding with a weighted steel surveyors'

tape or other steel or fiberglass measuring tape, weighted as needed. For shallow wells,

the electronic water-level probe may also be used as a measuring device. Procedures to

be followed are specified below.

1. For calibration, measure the distance between the zero mark on the end of the measuring

tape and the bottom of the weight to the nearest 0.1 foot at the beginning of each well

depth measurement activity day, and whenever the apparatus is altered.

2. Lower a weighted tape into the well until the tape becomes slack or there is a noticeable

decrease in weight, which indicates the bottom of the well. Care should be taken to lower

the tape slowly to avoid damage to the bottom of the well by the weight.

3. Raise the tape slowly until it just becomes taut, and with the tape in this fixed position,

note the tape reading opposite the MP to the nearest 0.1 foot.

4. Add the values from the distance from the end of the tape to the end of the weight

together, round this number to nearest 0.1 foot, and record the resulting value as "well

depth below MP" in the "Remarks" column of the Water Level Monitoring Record form.

OU3 SOP 13

Rev. No. 0

Date: September 26, 2007

Calibration of the electronic probe will be checked at regular intervals as part of regular

maintenance measuring the position of the electrode to check that the calibration marks on the

electronic probe correspond to those on the steel surveyors tape.

6.0 NAPL THICKNESS MEASUREMENT

Free product, or NAPL, thickness measurements will be made using a dual-phase probe. This

instrument is equipped with a sensor capable of detecting NAPL. The probe emits a steady

signal when in contact with NAPL and an intermittent signal when in contact with water. The

procedures outlined below will be followed in obtaining field measurements of NAPL thickness.

1. Caution will be exercised while opening and working around wells with known NAPL

contamination as explosive atmospheres may exist. Monitoring of the well

atmosphere will be conducted in accordance with the project-specific health and safety

plan.

2. Verify the well identification (ID) number. Check and ensure proper operation of

measuring equipment above ground. Prior to opening the well, don personal

protective equipment as required. Use caution when opening well cap.

3. If measuring LNAPL lower the probe slowly into the well. Upon contact with

LNAPL, the probe should emit a steady sound signal. Raise and lower the probe

slightly about the NAPL level a few times to accurately determine the point of contact.

Record the value determined to the nearest 0.01 foot from the surveyed reference point

on the well casing. If no reference is available, record in the logbook or on the field

form where the measurement was taken from (e.g., north side of inner casing).

4. With the buzzer still sounding advance the probe further down the well until the sound

signal changes from steady to intermittent. This should indicate the probe has come

into contact with water. Raise and lower the probe slightly about the LNAPL-water

OU3 SOP 13

Rev. No. 0

Date: September 26, 2007

Page 7 of 9

interface a few times to accurately determine the depth of the interface. Record the depth measurement from the tape. Values will be recorded to the nearest 0.01 feet from the MP. If no reference mark is present, record in the logbook or in the field form where the measurement was taken from (e.g., from the north side of the inner casing). The difference between the two measured values is the LNAPL thickness.

- 5. If determining DNAPL presence, lower the probe slowly into the well until an intermittent signal is emitted. Raise and lower the probe slightly above the water level a few times to determine the accurate point of contact. Record the measured depth to the nearest 0.01 feet from the surveyed reference point in the field logbook or on the field form. If no reference is available, record in the logbook or on the field form where the measurement was taken from (e.g., north side of inner casing).
- 6. Lower the probe slowly through the water column until a steady signal is emitted. Raise and lower the probe slightly about the DNAPL-water interface level a few times to accurately determine the depth of the interface. Record the DNAPL-water interface level to the nearest 0.01 feet from the surveyed reference point. If no reference is available, record in the logbook or on the field form where the measurement was taken from (e.g., north side of inner casing). The difference between the two values is the water thickness.
- 7. Record well number, top of casing elevation, and surface elevation if available.
- 8. Record the time and day of the measurement.
- 9. Some water-level measuring devices have marked metal or plastic bands clamped at intervals along the measuring line used for reference points to obtain depth measurements. The spacing and accuracy of these bands will be checked before each round of measurements because they may loosen and slide up or down the line, resulting in inaccurate reference points.

OU3 SOP 13

7.0 QUALITY ASSURANCE AND QUALITY CONTROL

All groundwater elevation measurements must be documented in the field logbooks and/or field

forms, including rationales deviations from this SOP. The Field Team Leader or designated QA

reviewer will check and verify that field documentation has been completed per this procedure

and other procedures referenced herein.

All equipment must be operated according to the manufacturer's specifications, including

calibration and maintenance. Calibration of the electronic probe will be checked at regular

intervals as part of regular maintenance measuring the position of the electrode to check that the

calibration marks on the electronic probe correspond to those on the steel surveyors tape.

8.0 DECONTAMINATION

All equipment used in the sampling process shall be decontaminated prior to field use and

between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall

don appropriate personal protective equipment as specified in the health and safety plan. Any

investigation-derived waste generated in the sampling process shall be managed in accordance

with the procedures outlined in SOP-12.

9.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Nielson, D.M., 1991. Practical Handbook of Ground-Water Monitoring, Lewis Publishers, Inc.,

Chelsea, MI.

U.S. Environmental Protection Agency (USEPA), 1986. RCRA Ground-Water Monitoring

Technical Enforcement Guidance Document, OSWER-9950.1, September 1986.

OU3 SOP 13

Rev. No. 0

Date: September 26, 2007

Date: June 30, 2008

SOP AMB-LIBBY-OU3 (Rev. 1)

Title: SAMPLING OF OUTDOOR AMBIENT AIR (Adapted from CDM-LIBBY-12 Rev1)

**APPROVALS:** 

**TEAM MEMBER** 

EPA Remedial Project Manager

SOP Author

SIGNATURE/TITLE

DATE

7/1/08

6/30/08

Revision Number	Date	Reason for Revision
0	09/26/2007	1000
1	06/30/2008	Remove OU3 phase specificity

SOP AMB-LIBBY-OU3 Rev. No. 1 Date June 30, 2008 Page 1 of 7

#### 1.0 OBJECTIVE

The objective of this standard operating procedure (SOP) is to establish the baseline requirements, procedures, and responsibilities inherent to the collection of outdoor ambient air samples. Air samples will be collected as part of the outdoor ambient air program conducted in accordance with the applicable Libby Operable Unit 3 (OU3) Sampling and Analysis Plan (SAP).

#### 2.0 RESPONSIBILITIES

All staff with responsibility for the collection of outdoor ambient air samples are responsible for understanding and implementing the requirements contained herein as well as other related project-specific requirements.

**Team Leader** – The team leader is responsible for communication with EPA regarding status and progress of the sampling event and providing support to field team staff to ensure all necessary resources are available for implementation of the ambient air program.

**Field Team Leader (FTL)** – The FTL is responsible for ensuring that the specifics related to the collection of outdoor ambient air sampling described in this procedure are followed by all staff and that all quality assurance/quality control procedures related to this program are implemented.

**Field Team Members** – The field team members are responsible collection and documentation of samples as described in the SAP and this SOP.

### 3.0 REQUIRED EQUIPMENT

The following is a general list of equipment that may be used:

- Sampling pump The selected sampling pump will be capable of a flow-rate and pumping times sufficient to achieve the desired air sample volume. The sampling pump will provide a non-fluctuating air-flow through the filter, and will maintain the initial volume flow-rate to within ±10% throughout the sampling period.
- Transmission electron microscopy (TEM) sample cassettes commercially available, 25-millimeter (mm), three-piece cassette with a 50mm electronically conductive extension cowl loaded with a 0.8 micrometer (μm) mixed cellulous ester (MCE) filter.
- Pump housings –Weatherproof enclosure to house pump and batteries.
- Stands metal fenceposts to hold sample cassettes at the desired height (6 feet) will be used
  to support the sample cassette in order to isolate the sample from the vibrations of the
  sampling pump.
- Inert tubing Tygon tubing used in the sampling train to connect the outflow end of the sample cassette to the sampling pump. Tubing has a 1/4" inner diameter and 7/16" outer diameter.

SOP AMB-LIBBY-OU3 Rev. No. 1 Date June 30, 2008 Page 2 of 7

- Rotameter A rotameter calibrated such that the operator can measure flow rates to  $\pm 5\%$  accuracy at the expected sampling flow rate.
- Field sample data sheets (FSDS) specific data related to the collection of each sample will be recorded on a OU3-specific stationary ambient air FSDS. This sheet will contain all relevant information regarding equipment used, flow rates, and collection times.
- Field logbook
- Plastic bags
- Sample labels
- Clear packaging tape
- Ink pen

# **4.0 PROCEDURES**

# 4.1 Selection of Air Volumes and Flow Rates

Samples collected as part of the outdoor ambient air monitoring program are to be collected over a 5-day sampling period. This represents collection duration of 120 hours or 7,200 minutes. The target volume of air to be collected for each sample will be 14,000 liters for the collection period. As a result, samples will be collected at a flow rate of 2.0 liters/minute, which will result in a sample volume of approximately 14,400 liters.

In no event shall a sample be collected at a flow rate lower than 0.92 L/min, since the linear flow velocity would fall below 4 cm/sec, which is the minimum velocity specified by International Organization for Standardization (ISO) method 10312.

As samples are initially collected during this program and analyzed, flow rates and sample times may be adjusted to ensure the sample filter has proper loading for the required analytical analysis and sensitivity goals.

# 4.2 Calibration Procedures and Flow Rate Adjustments

Each sampling pump will be calibrated before the start of each ambient air sample collection cycle. This is to ensure that each sampling pump is measuring the flow rate or volume of air correctly.

Calibration of Rotameter with an Electronic Calibrator

Rotameters used for pump calibration are calibrated to a primary flow standard. Procedures for rotameter calibration with the primary flow standard meter are as follows:

1. If the electronic calibrator does not automatically adjust to account for temperature and pressure changes, obtain the actual temperature and pressure in Libby, MT from the local National Oceanic and Atmospheric Administration (NOAA) weather station or

SOP AMB-LIBBY-OU3 Rev. No. 0 Date September 26, 2007 Page 3 of 7 from temperature and weather reference centers. Record actual temperature and pressure in the fields provided on the Precision Rotameter Calibration Data Sheet (Attachment 1).

- 2. Set up the calibration train as shown in EPA SOP #2015 Figure 4 (Attachment 2) with the sampling pump, rotameter, and primary flow meter.
- 3. Hold the rotameter as vertical as possible.
- 4. Turn the primary flow standard and sampling pump on.
- 5. Adjust the pump until the desired flow rate is attained.
- 6. Calibrate rotameter to desired ball reading, as read from the middle of the flow ball. Record value in the Ball Reading column on the rotameter calibration data sheet.
- 7. Check adjusted flow rate of sample pump to the primary flow standard. Ten repetitive flow measurements will be averaged and that result recorded in the flow rate column for the selected interval.
- 8. Repeat this process at 10 intervals over the range of the precision rotameter.
- 9. Input data into rotameter calculation sheet to generate the corrected flow rate.

# Calibration of Sampling Pump with a Rotameter

Prior to sample collection, each sampling pump will be calibrated with a rotameter that has been calibrated as described in Section 4.2.1. The procedures used for sampling pump calibration are as follows:

- 1. Set up the calibration chain as shown in EPA SOP #2015, Figure 5 (Attachment 2) using a rotameter, sampling pump and a representative sample cassette. The sample cassette to be used for sampling is installed between the pump and the calibrator.
- 2. To set up the calibration train, attach one end of tubing to the sample cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the sample cassette cap to the rotameter.
- 3. Hold the rotameter as vertical as possible.
- 4. Turn the sampling pump on.
- 5. Adjust the sampling pump until the middle of the float ball on the rotameter is lined up with the pre-calibrated flow rate value.

Each rotameter used for field calibration will be transported to and from each sampling location in a sealed zip-top plastic bag. The sample cassette cap and attached tubing for rotameter checks will be stored in the site enclosure in a plastic bag.

SOP AMB-LIBBY-OU3 Rev. No. 1 Date June 30, 2008 Page 4 of 7

# Adjustment of Flow Rates During Flow Checks

Flow checks will be performed daily. During these checks, flow rates will be adjusted back to the target rate of 2.0 L/min. Adjustment of flow rates during flow checks will be performed as described below and as the last action before leaving a sampling location anytime the sampling pump is moved. Should the flow rate change from the target flow rate, the following procedure will be used to make the adjustment:

- 1. Connect the rotameter as described in Section 4.2.2 steps 1 to 3.
- 2. Record the observed flow rate and time of observation.
- 3. Adjust the flow rate to the target flow of 2.0 L/min.

Attachment 3 illustrates the volume tracking spreadsheet that will be used in the field to determine the time required for sample collection. To track the scheduling of calibration checks, the field teams will use the Calibration Check Schedule Worksheet. An example of this worksheet is included as Attachment 4. Copies of all volume tracking spreadsheets and Calibration Check Schedule Worksheets will be provided to the project data manager at the conclusion of each sampling event. Electronic copies are suitable and will be placed in the project-specific FTP site within one week from completion of each sampling event.

# 4.3 Outdoor Ambient Air Sample Collection Procedures

Selection of Outdoor Ambient Air Sampling Locations

The position of each sampling location will be fixed; the sample will be collected from the same location for the duration of the outdoor ambient air sampling program. GPS points will be collected for the location of each station. The location should be placed to minimize interference from large trees and road dust.

# Sampling Protocol

Each outdoor ambient air sample will be collected, after pump calibration, according to the following procedures:

- 1. Place a sample label on the sample cassette indicating a unique Index ID.
- 2. Set up the sampling train; attach the air intake hose to the sample cassette base. The sample cassette will be positioned such that it is held facing downwards at an angle equal to or less than 45° from horizontal. Set the sample cassette to a height of 6 feet above ground surface. Remove the sample cassette cap.
- 3. Complete the upper portion of the FSDS form including: station information, Index ID, collection height, location description, QC type, cassette lot number, pump number, flow meter number, pump start date and time, and starting counter.
- 4. Turn the sampling pump on.
- 5. Check the sampling pump daily and adjust the flow rate, as needed.

SOP AMB-LIBBY-OU3 Rev. No. 0 Date September 26, 2007 Page 5 of 7

- 6. At the end of the sampling period, orient the sample cassette up, do not remove the sampling cassette from the sampling train. Record the ending flow rate.
- 7. Turn the pump off. Record the pump stop date and time.
- 8. Remove the tubing from the sample cassette. Still holding the sample cassette upright, replace the inlet plug on the sample cassette cap and the outlet plug on the sample cassette base. Note: Do not put sample cassettes in shirt or coat pockets as the filter can pick up fibers.
- 9. Place each sample cassette in a plastic sample bag. Each bag should be marked with the same Index ID as the sample cassette. Place clear packaging tape over the sample identification label.
- 10. Transport the sample cassettes to the sample coordination area and provide the sample coordinator with the appropriate documentation with the samples.

# Pump Failure Procedures

If a sampling pump faults prior to the total desired run time, the following procedures should be used:

- 1. Record the time of the observed pump fault in the field notes.
- 2. Record the stop time as the time of the last field flow check.
- 3. Record the ending flow rate as the rate observed at the time of the last field flow check.
- 4. Turn the sampling pump back on and calibrate as required (Section 4.2.2).
- 5. Restart sample collection.
- 6. Note the pump fault on the FSDS form.
- 7. Use pump fault time and flow information, if it can be retrieved.

If a pump fault occurs a second time, sampling shall be terminated, and the sample will be archived.

# 4.4 Quality Assurance/Quality Control

# Equipment Maintenance

The manufacturers' instructions regarding operating procedures and maintenance will be reviewed prior to equipment use. Equipment and instrumentation will be utilized in accordance with manufactures instructions.

# Collection of Field Quality Control Samples

Field quality control (QC) samples will consist of three types: lot blanks, field blanks, and colocated samples. Field QC samples will be collected at the frequency specified in the applicable OU3 SAP.

### 4.5 Documentation

Documentation of outdoor ambient air sampling will be recorded in three main locations: field logbooks, field sample data sheets, and the Daily Observation/Impact memorandum. Each of these is discussed below.

# Logbooks

Documentation of field activities conducted as part of this program will be recorded in logbooks maintained specifically for this sampling program. The procedures specified in OU3 SOP No. 9 will be followed for logbook records.

# Field Sample Data Sheets

Detailed sampling information will be recorded for each sample on an OU3-specific stationary ambient air FSDS (provided in SOP No. 9, Attachment 1).

# Daily Observation/Impact Memorandum

For each day that outdoor ambient air samples are collected a Daily Observation/Impact Memorandum (DOI) will be completed. An example of the DOI is included in Attachment 5.

Information to be recorded will include the following: general weather conditions (including if there is any visible frost on the ground/sample cassette and if there is existing snow cover on the ground), location of all removal and remedial actions being conducted, other observations that could affect sample results, and any equipment issues.

# **5.0 REFERENCES**

International Organization of Standardization (ISO). 1995. Ambient Air – Determination of Asbestos Fibers – Direct Transfer Transmission Electron Microscopy Method. ISO 10312:1995(E).

# SOP AMB-LIBBY-OU3

# ATTACHMENT 1

PRECISION ROTAMETER CALIBRATION DATA SHEET

Sheet	No.:	F1-		
-------	------	-----	--	--

# LIBBY OU3 PHASE 1 Precision Rotometer Calibration Data Sheet

alibration Date:			Calibration By:					
Rotometer ID:		Primary Standard ID:						
	·	Actual Pressure (in. Hg):						
		Flow Rate = X <sub>1</sub> (L/min)						
		<u> </u>						
					•			
					4			
		·						
:								
			•					
			Primary Standard ID: Actual Pressure (in. Hg)  Flow Rate = X <sub>1</sub> (L/min)	Primary Standard ID:Actual Pressure (in. Hg): Flow Rate = X <sub>1</sub> (L/min)	Primary Standard ID:Actual Pressure (in. Hg):Flow Rate = X <sub>1</sub> (L/min)			

### Rotometer Calibration Procedure:

- 1. Obtain the actual temperature and actual pressure in Libby, MT from the MET station. Record the actual temperature and actual pressure in the fields provided above.
- 2. Calibrated rotometer to desired ball reading with a sampling pump and cassette in-line. Cassette must be the same type and from the same lot of cassettes that will be used for sampling. Record value in Ball Reading column.
- 3. Check adjusted flowrate of sample pump to the Dry Cal flow calibrator primary flow standard. 10 repetitive flow measurements will be averaged and that result recorded in the Flow Rate column for the selected interval.
- 4. Repeat this process at 10 intervals over the range of the precision rotometer.
- 5. Input data into rotometer calculation sheet to generate the corrected flow rate.

# **ROTAMETER CALIBRATION CALCULATIONS**

Rotameter #: Calibration Date Primary Standard ID: Air Pump(s) ID:



X<sub>1</sub> = Actual Flow Rate (lpm) Y = Ball Reading (Center)

y = mx + b

m = x coef. = slope

b = const.

 $X_1$ 

Regression Output:

Constant #DIV/0! Std Err of Y Est #DIV/0! R Squared #DIV/0!

No. of Observations

X Coefficient(s)

#DIV/0!

**Corrected Data** 

X<sub>2</sub> = Corrected Flow Rate (lpm)

Y = Ball Reading

Actual Temperature (°F) =

Actual Barametric Pres. (in Hg) =

 $X_2$ 

0.00 0.00 0.00 0.00 0.00 0.00

0.00 0.00 0.00

**Regression Output:** 

Constant #DIV/0! Std Err of Y Est #DIV/0! R Squared #DIV/0I No. of Observations

X Coefficient(s)

#DIV/0!

10

# SOP AMB-LIBBY-OU3

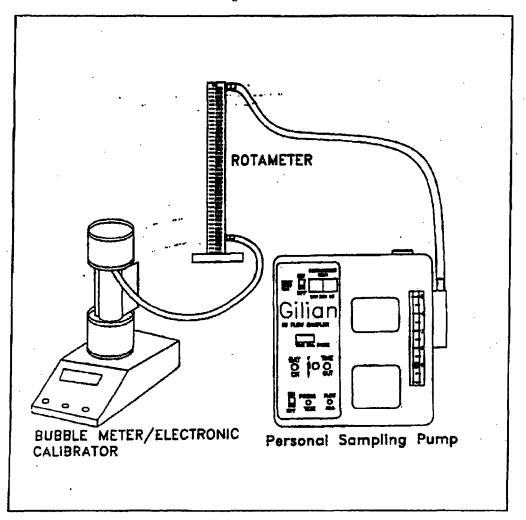
**ATTACHMENT 2** 

CALIBRATION TRAIN FIGURES (from EPA SOP #2015)

# APPENDIX B

# Figures

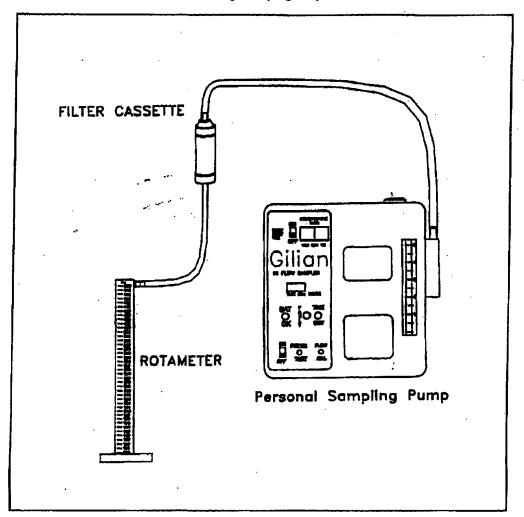
FIGURE 4. Calibrating a Rotameter with a Bubble Meter



# APPENDIX B

Figures

FIGURE 5. Calibrating a Sampling Pump with a Rotameter



# SOP AMB-LIBBY-OU3

# **ATTACHMENT 3**

VOLUME TRACKING SPREADSHEET EXAMPLE

# Poor Quality Source Document

The following document images have been scanned from the best available source copy.

To view the actual hard copy, contact the Region VIII Records Center at (303) 312-6473.

# LIBBY OU3, PHASE I SAMPLING: AIR MONITG. FOLUME CALCULATOR SPREADSHEET (v1)

		amina	Pump		Ştar	Informati	on	Day 1	Check	Day 2	Check	Day 3	Check -	Day 4	Check		Stop Infor	matlon		Data Entry	Total
Index ID	Pump ID	Flow Meter	(Yes/No)	Pump Comments	Date (mm/dd/w)	Time (bh:mm)	Counter	Time (bb:mm)	Flow.	Time	Flow	Time	Flow (L/min)	Time	Flow	Date (mm/dd/w)	Time (bb:mm)	Counter	Flow (L/min)	Checked by	Total Sample Volume (L)
Example P1-00001	(e 62862)	Source:		Pump Comments No issues encountered	(68/61/69)	8 00	256.7	9102	100	H 54		0.76		13.03	20	08/08/07	1011	LA SZ B			16,827
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# SOP AMB-LIBBY-OU3

# ATTACHMENT 4

CALIBRATION CHECK SCHEDULE SPREADSHEET EXAMPLE

# LIBBY OU3 AMBIENT AIR CALIBRATION CHECK SCHEDULE

		•	
Week No.	Dates:	 	

A-	·1	A-		A-		A-4		
Time Sched	Time Complete							
							<del></del>	
			<del></del>		<del></del>	 	·	

A-	5	A-	-6	A-	.7	A-8.		
Time Sched	Time Complete							
_								
·								

# SOP AMB-LIBBY-OU3

# **ATTACHMENT 5**

DAILY OBSERVATION/IMPACT MEMORANDUM SPREADSHEET EXAMPLE

# LIBBY OU3 AMBIENT AIR SAMPLING PROGRAM DAILY IMPACT/OBSERVATION MEMORANDUM

This report represents a summary of observations made during the say that could potentially impact the results of samples collected as part of the ambient air sampling program as described in AMB-LIBBY-OU3: Collection of Outdoor Ambient Air Samples.

of Outdoor Ambient Air Samples.		
Date: From:		
Weather Conditions: describe the ger	neral weather conditions for the da	ate of this memo.
OBSERVED ACTIVITIES: describe observed, city maintenance).	served activities that could effect	sample results (wildfires, street
Potentially Impacted Sample Locations	Description of Activity	Estimated Proximity to Sample Location
EQUIPMENT ISSUES: describe all equ	ipment issues that could affect s	
Potentially Impacted Sample Locations	Issue	Description of Actions Taken to Rectify Issues
DEVIATIONS FROM GUIDANCE DOC ambient air program.	UMENT: describe previously und	
Potentially impacted Sample Locations	Describe Deviation	List Mod Form Number Completed
	•	Í

Date: June 30, 2008

Libby Well Redevelop (Rev. 0)

Title: WELL REDEVELOPMENT

APPROVALS:

**TEAM MEMBER** 

EPA Remedial Project Manager

**SOP Author** 

SIGNATURE/ITILE

6-30-08

Revision Number	Date	Reason for Revision
0	09/26/2007	-

Libby Well Redevelop Rev. No. 0 Date: June 30, 2008

# STANDARD OPERATING PROCEDURE

# WELL REDEVELOPMENT

**JUNE 2008** 

Project No.:

1005439.080801

Prepared by:
MWH Americas, Inc.
10619 South Jordan Gateway, Suite 100
Salt Lake City, Utah 84095

# TABLE OF CONTENTS

			PAGE NO.
1.0	INT	RODUCTION	1-1
	1.1	Sequence of Activities for Performing Well Redevelopment	1-2
	1.2	Definitions	1-3
2.0	GEI	NERAL REQUIREMENTS	2-1
	2.1	Health and Safety Considerations	2-1
	2.2	Field Documentation	2-1
	2.3	Investigation-Derived Waste	2-2
	2.4	Decontamination	2-2
3.0	PRO	OCEDURES FOR WELL REDEVELOPMENT	3-1
	3.1	Pre-Redevelopment Activities	3-1
		3.1.1 Pre-Redevelopment Static Water-Level Measurement	3-1
		3.1.2 Remove Pumping Equipment	3-1
		3.1.3 Pre-Redevelopment Total Well-Depth Measurement	3-1
		3.1.4 Pre-Redevelopment Hydraulic Testing	3-2
		3.1.5 Water-Level Recovery	3-3
	3.2	Mechanical Well Redevelopment	3-3
	3.3	Post-Redevelopment Activities	3-6
		3.5.1 Post-Redevelopment Total Well-Depth Measurement	3-6
		3.5.2 Post-Redevelopment Hydraulic Testing	3-6
REFI	ERE	NCES	R-1

# LIST OF ATTACHMENTS

ATTACHMENT A Specific Capacity Testing Data Sheet ATTACHMENT B Borehole Purge Volume Calculations

# LIST OF ACRONYMS

CFR Code of Federal Regulations

BD borehole diameter

gpm gallons per minute

gpm/ft gallons per minute per foot

ID inner diameter

IDW investigation-derived waste

NTU nephelometric turbidity unit

SC standing water column

SOP Standard Operating Procedure

SL screen length SpC specific capacity

TD total depth

WL water level

# 1.0 INTRODUCTION

This Standard Operating Procedure (SOP) describes the general procedures that will be followed during redevelopment of groundwater wells at Operable Unit 3 (OU 3) of the former W.R. Grace Mine site located near Libby, Montana. The objective of well redevelopment is to remove loose sediments and encrustation material that may affect the representativeness of groundwater samples collected from the wells. This SOP describes procedures for the removal of fine-grained material that may be plugging the well, filter-pack, and the natural formation in the immediate vicinity of the screened interval. The procedures outlined in this document are not intended to address wells that are fouled by chemical encrustation or biofouling.

Well redevelopment as defined in this SOP involves mechanical processes only. Mechanical processes involve brushing, surging, bailing, and possibly pumping the well to remove fine-grained material from the well, screen, filter-pack, and the natural formation in the immediate vicinity of the screened interval. The procedures described in this SOP are based on industry standards for redeveloping groundwater wells as well as other types of wells (e.g. extraction and production wells). This SOP is intended to provide procedural guidelines that allow flexibility based on well construction, real-time field observations, and collaboration between the project team (e.g., the W.R. Grace /Remedium Group Project Manager, the MWH Project Manager, experienced MWH hydrogeologists, and an experienced well-service contractor).

# 1.1 SEQUENCE OF ACTIVITIES FOR PERFORMING WELL REDEVELOPMENT

The general sequence of activities to be followed during well redevelopment are as follows:

- 1) Measure initial static water-level.
- 2) Remove pumping system, if applicable.
- 3) Measure pre-redevelopment total well depth.

- 4) Measure water-quality parameters (e.g. pH, specific conductivity, temperature, dissolved oxygen and turbidity).
- 5) Perform pre-rehabilitation short-term specific capacity (SpC) test using a dedicated bailer or clean submersible pump to evaluate the general hydraulic conductivity of the well prior to development.
- 6) Perform mechanical redevelopment.
- 7) Measure water-quality parameters.
- 8) Perform additional mechanical redevelopment.
- 9) Measure water-quality parameters.
- 10) Perform post-rehabilitation short-term SpC test using a dedicated bailer or clean non-dedicated submersible pump test the general hydraulic conductivity of the well after development.

Detailed procedures for well redevelopment are provided in the following sections of this SOP.

# 1.2 DEFINITIONS

The following definitions are used throughout the remainder of this SOP:

- Bailer A long, narrow bucket-like device with an open top and a check valve at the bottom that is used to remove water and/or sediments from the well.
- Filter-Pack Uniform, clean sand or fine gravel that is placed in the annulus
  of the well (between the borehole wall and the well screen) to stabilize the
  adjacent formation and also prevent formation material from entering through
  the well screen.
- Flow Rate The rate at which water is being pumped out of a well; usually measured in gallons per minute (gpm).

• Surge Block – A surge block is a downhole tool comprising a shaft with a larger diameter rubber packer or "squeegee." The packer diameter is slightly larger than the inside diameter of the well casing and screen. The surge block is used in an up and down motion in the screened section to gently force water into and out of the filter pack and adjacent formation. The packer typically has a relief hole or valve to limit pressure to the filter pack – the relief valve is particularly important when surging wells that are screened adjacent to low permeability formations. The surge block also can be equipped with a circular hard plastic or wire brush placed below the packer to loosen scale or biofilm from the well screen.

# 2.0 GENERAL REQUIREMENTS

This section describes the general procedures for redeveloping wells.

# 2.1 HEALTH AND SAFETY CONSIDERATIONS

The health and safety of all contracted employees is the responsibility of the contractor or subcontractor and all appropriate health and safety equipment required for the work (e.g., personal protective equipment) will be provided by the contractor. The contractor is responsible for having copies of the material safety data sheets (MSDS) for any chemicals brought to the site and for training contractor employees concerning hazard communication (29 CFR 1910.1200). A documented tailgate safety meeting will also be performed daily, and will be attended by all on-site personnel.

# 2.2 FIELD DOCUMENTATION

Field details of well redevelopment will be recorded in a field logbook and on a designated field form. The following information will be documented during the well redevelopment process:

- Well identification
- Date and time of field activities
- Measured well depth (pre- and post-redevelopment)
- Water levels (pre- and post-redevelopment)
- Pumping rate and water-level drawdown during SpC testing
- Periodic water-quality parameters during development
- Type of equipment used
- Total volume of water removed
- Purge water handling and disposal information

# 2.3 INVESTIGATION-DERIVED WASTE

MWH anticipates that investigation-derived waste generated during well redevelopment activities at OU3 will be limited to spent PPE. Most of the wells at OU 3 were production wells and there is no reason to assume that any of the wells were contaminated. For redevelopment of the OU 3 wells, it is assumed that the purge water can be safely discharged to the ground surface, away from the well head. Water-quality parameters (e.g. pH, specific conductivity, temperature, dissolved oxygen and turbidity) will be measured in the field. If any indications of contamination are detected (high or low pH, odors, colors or sheens) purging will cease immediately and the purge water will be collected for characterization and proper disposal. Purge water volumes will be recorded in the field logbook.

# 2.4 DECONTAMINATION

It is important that every effort be made to avoid contamination or cross-contamination of the wells. All down hole equipment including bailers, surge blocks and pumps will be steam cleaned or thoroughly hand washed using an anionic detergent (e.g., Alconox or Liquinox) and distilled water or water from an approved source before the first use and after redevelopment activities have been completed at each well.

# 3.0 PROCEDURES FOR WELL REDEVELOPMENT

This section of the SOP describes the general procedures for mechanically redeveloping wells.

# 3.1 PRE-REDEVELOPMENT ACTIVITIES

# 3.1.1 Pre-Redevelopment Static Water-Level Measurement

The depth to water level will be measured to the nearest 0.01 of a foot using a clean, electric water-level indicator. The depth to water will be measured at the designated measuring point (usually the north side of the top of casing) each time a water level is collected; this will ensure consistent measurement data throughout the redevelopment process. The pre-redevelopment depth-to-water measurement will be taken prior to removing any pump system in the well.

# 3.1.2 Removal of Pump System

If the well is equipped with a pump, drop-pipe, wires, tubing or other equipment, they will be removed by lifting by the well-service rig.

# 3.1.3 Pre-Redevelopment Total Well-Depth Measurement

The well depth will be measured using the electric water-level indicator (to the nearest 0.01 of a foot). The well depth will be measured at the same designated measuring point as the static water level each time a measurement is collected; this will ensure consistent data throughout the redevelopment process.

# 3.1.4 Pre-Redevelopment Hydraulic Testing

Hydraulic testing will consist of a simple short-term specific capacity (SpC) test to measure flow rate and associated drawdown in gallons-per-minute per foot (gpm/ft). These data will be useful for evaluating the immediate benefits of the redevelopment

activities and will be useful for establishing a baseline for this parameter. The SpC tests will be performed with a decontaminated submersible pump or disposable bailer. The test will be limited to establishing a sustainable pumping rate and associated drawdown over a 60-minute pumping period for a simple comparison between pre-and post-redevelopment test results.

If a submersible pump is used it will be set in the screened section of the well, approximately 2 feet above the bottom of the well, for the duration of the test. The initial pumping will be at a low rate (e.g., 1 gpm) and will be adjusted accordingly to maintain a quasi-steady-state water level near, but above the pump intake. Ideally, a submersible pump will be used for the SpC testing and the flow rate will be displayed on the controller. In small-diameter wells, a dedicated disposable bailer may be used instead of a submersible pump.

During pumping, water-level measurements will be collected manually to the nearest 0.01 foot with an electric water-level meter to monitor the drawdown of the groundwater. The time, depth-to-water, and pumping rate in gpm will be recorded at 5 minute intervals on the attached Specific Capacity Testing Data Sheet. The stabilized pumping rate will be divided by the stabilized drawdown (pre-test water level minus the stabilized water level) to calculate the SpC in gpm/ft. The stabilized pumping rate established during the pre-redevelopment test will be the initial flow rate during the post-redevelopment SpC test.

# 3.1.5 Water-Level Recovery

After hydraulic testing, the water level in the well will be allowed to recover to approximate static equilibrium prior to performing the initial mechanical redevelopment described below. If the rate of change between water-level readings is equal to or less than 0.02 foot per hour (about 0.5 feet per day), then it can be assumed that the water level recovery has reached static equilibrium. However, if the rate of change between readings is large (i.e., greater than 0.02 foot per hour), then further time will be allowed for aquifer recovery until rates reach a level equal to or less than 0.02 foot per hour.

# 3.2 MECHANICAL WELL REDEVELOPMENT

Mechanical redevelopment will consist of brushing and/or surging the screened section of the well followed by bailing and/or pumping to remove scale, sediment, and other debris dislodged from the well. The brushing and surging process may be completed simultaneously if a brush is built onto the surge block. The brush and/or surge block will be lowered and raised repeatedly to perform the brushing and surging action.

Each 10-foot section of well screen will be brushed and/or surged separately for an approximate duration of 15 minutes, followed by bailing and or pumping to remove the dislodged materials. This process will continue until the entire section of well screen has been brushed and surged. Brushing and/or surging will continue until there is a noticeable decrease in the amount of scale, sediment, and debris being bailed or pumped from the well. However, it will be recognized that the brushing/surging process can damage the well if the screen is broken or the tools are of improper size. If it appears that excessive amounts of filter-pack are being drawn into the well, it may be prudent to reduce the brushing/surging efforts to minimize further damage to a broken well screen or removal of the filter-pack. These types of observations and revised procedures will be clearly noted in the field logs.

# 3.3 MEASUREMENT OF WATER-OUALITY PARAMETERS

- Monitor groundwater quality parameters including pH, specific conductivity, temperature, and turbidity. These parameters will be measured at the beginning of well redevelopment and after evacuation of each borehole volume.
- 2) A minimum of three borehole volumes will be removed from the wells by bailing and pumping.
- 3) Water collected from the well will be considered representative of formation water when water-quality parameter measurements satisfy the following criteria:

- pH  $\pm 0.5$  of pre-redevelopment measurements
- Specific Conductivity  $\pm 10\%$  of pre-redevelopment measurements
- Temperature  $\pm 1^{\circ}$  of pre-redevelopment measurements
- Turbidity < 5 nephelometric turbidity units (NTUs) preferable; up to 30</li>
   NTUs is acceptable.

# 3.5 POST-REDEVELOPMENT ACTIVITIES

# 3.5.1 Post-Redevelopment Total Well-Depth Measurement

The well depth will be measured using an electric water-level indicator (to the nearest 0.01 of a foot) immediately following the mechanical redevelopment activities. The post-redevelopment well depth should be very similar to the well depth noted on the well completion log (if available). No debris or sediment should remain in the well bottom following redevelopment activities.

# 3.5.2 Post-Redevelopment Hydraulic Testing

The simple short-term SpC test described above in Section 3.1.4 will be repeated after well redevelopment to measure flow rate and associated drawdown in gpm/ft. These results will be compared to the pre-redevelopment SpC results to evaluate the immediate benefits of the redevelopment activities and to establish a post-redevelopment SpC baseline for each well.

REFERENCES	
Code of Federal Regulations (CFR). The OSHA Hazard Com No. 1910, Occupational Safety and Health Standard Hazardous Substances, 29 CFR 1910.1200.	munication Standard - Part ds, Subpart Z Toxic and
	·

# ATTACHMENT A SPECIFIC CAPACITY TESTING DATA SHEET

# ATTACHMENT B BOREHOLE PURGE VOLUME CALCULATIONS

# ATTACHMENT B

# BOREHOLE PURGE VOLUME CALCULATIONS

The following information is used to calculate casing, annular, and total borehole volumes:

- 1) Total depth of well (TD)
- 2) Measured static water level (WL)
  - 3. Screen length (SL)
- 3) Well casing inner diameter (ID)
- 4) Borehole diameter (BD)
- 5) If the standing water column (SC) is longer then the screen length, the length of filter pack installed above the screen needs to be included in the borehole volume calculation.

**Total Borehole Volume:** 

$$V_t = (V_c + V_a n) \times 7.48 \text{ gal/ft}^3$$

where:

V<sub>t</sub> = Total purge volume (gals)

 $V_c = Volume of water in well casing (ft^3)$ 

 $V_a = Volume of water in well annulus (ft<sup>3</sup>)$ 

n = Estimated porosity of sand pack (usually 30%)

**Casing Volume:** 

$$Vc = \pi r_1^2 h_1$$

where:

 $V_c = Casing volume (ft^3)$ 

 $r_1$  = Radius of well casing (ft)

 $h_1$  = Height of water column (i.e., total well depth minus

static water level depth) (ft)

$$Va = \pi (r_2^2 - r_1^2) h_2$$

where:

 $V_a = Annular volume (ft^3)$ 

 $r_2$  = Radius of borehole (ft)

 $r_1 = Radius of well casing (ft)$ 

 $h_2$  = Total vertical saturated thickness of sand pack (ft).

# ATTACHMENT B LIBBY LABORATORY MODIFICATIONS FOR TEM ANALYSES



# **Request for Modification**

#### To Laboratory Activities LB-000016

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs

Individual Lab Applicable forms - copies to: EPA, Volpe, CDM-Denver, Initiating Lab Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM 05755-95, EPA/540/2-90/005a, Other. Title: President Requester: Jeanne Orr Company: Reservoirs Environmental, Inc. Date: December 2, 2002 Description of Modification: Permanent modifications and clarifications to the Transmission Electron Microscopy analysis of air samples using ISO 10312. The purpose of the attached is to document permanent historic modifications & clarifications. Reason for Modification: To optimize the efficiency of air sample analysis and to provide consistency in analytical procedures and data recording in the project laboratories. Potential Implications of this Modification: Modifications reflect changes necessary to clarify ISO requirements in relation to project-specific issues. No negative implications to these modifications are anticipated. Positive implications are consistency in procedures between and within project laboratories and documentation of those procedures. Laboratory Applicability (circle one): All Individual(s) Duration of Modification (circle one): Temporary Date(s): Analytical Batch ID: Temporary Modification Forms - Attach legible copies of approved form w/ all associated raw data packages Permanent (complete Proposed Modification Section) Effective Date: HISTORIC Permanent Modification Forms - Maintain legible copies of approved form in a binder that can be accessed by TEM analysts. Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): Please see the attached for the description of the TEM-ISO clarifications/modifications **Technical Review:** (Laboratory Manager or de

Date: 4 April 2003

Title: Projet Chenust Date: 3 April 2003

Approved By: (USEPA: Mary
Devision-Modification for TEM ISO

Project Review and Approval: (Volpe: Mark Reney)

#### 1. Modification:

The ISO method requirement is if the specimen grid exhibits more than approximately 10% obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. A rejection criteria of >25% obscuration and <50% intact grid openings will be used for this project. The 25 % overload criteria resulted from various communications that took place 29 December 1999 between EPA Region 8, Camp Dresser McKee, Volpe Center, and Reservoirs.

#### 2. Modification:

ISO 10312 is a direct preparation method. If samples are visibly overloaded or contain loose debris and they have not been previously analyzed (the filter is whole) they will be prepared indirectly according to procedures described in ASTM D5755-95. If the sample has been previously analyzed or rejected in the microscope (section removed from the filter), prepare the sample indirectly according to EPA/540/2-90/005a by plasma ashing a portion of the original filter and depositing an aliquot on a secondary filter. Secondary filters will be analyzed according to the ISO counting rules for this project. Calculations are modified to contain a dilution factor. This indirect preparation procedure is embraced to enable the capture of data from samples that otherwise would be rejected.

#### 3. Clarification:

Stopping rules for ISO analyses are completion of the grid opening on which the 100<sup>th</sup> asbestos structure has been recorded, or a minimum of four grid openings. For this project, a maximum of ten grid openings will be read unless specifically instructed otherwise.

If abundant chrysotile is present, the chrysotile count may be terminated at the end of the grid opening where the 100th chrysotile structure is counted. The analysis will continue recording amphibole fibers only until the remaining grid openings to be analyzed are completed. The grid opening location designation will be followed by a "\*" to indicate the grid openings where only amphibole asbestos was recorded, i.e. K6\*.

This clarification in structure counting and recording is to provide consistency in analytical procedures and data recording in the project laboratories.

#### 4. Modifications and clarifications: Structure counting and recording

- a. Modification: Non-asbestos structures are not being recorded. This project-specific modification stems from our need only to quantify contaminants of concern: the asbestos levels at a given sample location.
- b. Modification: The overall dimensions of disperse clusters (CD) and disperse matrices (MD) will not be recorded in two perpendicular directions. The matrix type and individual structures associated with the matrix or cluster will be recorded as described in the ISO method.
- c. Modification: Structures that intersect a non-countable grid bar will be recorded on the count sheet but excluded from the structure density and concentration calculations.
- d. Modification: If a structure originates in one grid opening and extends into an adjacent grid opening, providing that it does not intersect a non-counting grid bar, the entire length of the fiber is recorded.
- e. Clarification: If a structure intersects both a countable and a non-countable grid bar, the observed length of the structure will be recorded.

These modifications and clarifications in structure counting and recording are to provide consistency in analytical procedures and data recording in the project laboratories.

# Mahoney, Ron

From: Sent:

Reney, Mark [RANEY@VOLPE.DOT.GOV] Tuesday, April 22, 2003 11:09 AM 'Mahoney, Ron'

To: Subject:

FW: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017







```
> ----Original Message----
> From: Raney, Mark
> Sent: Friday, April 04, 2003 9:31 AM
> To: 'Backham, Richard'; 'Goldade.mary@EPAmail.epa.gov'; 'mgoklade@peakpeak.com'
> Co: Autlo, Anni
 > Subject: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017

    Volpe provides approval to revised MODs LB-000015, LB-000016, & LB-000017 as attached. The attached MODs include the following changes to the previous versions (received 4/1/03).

    The date indicated in the "Effective Date" field was removed and replaced with "HISTORIC"
    Under the "Description of Modification" section the following sentence was added "The purpose of the attached is to document permanent historic modifications & clarifications."

 If you have any questions as to these changes or the reason behind them let me know. Please proceed with distribution of the accepted versions of the attached for final hardcopy signature.
 >> <<LB-000015_rev (MR 4-4-03 email).doc>>>> <<LB-000018_rev (MR 4-4-03 email).doc>>>> <<LB-000017_rev
 (MR 4-4-03 email).doc>>
> ----Original Message----
> From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
> Sent: Tuesday, April 01, 2003 10:47 AM
> To: 'Goldade.mary@EPAmail.epa.gov'; 'RANEY@VOLPE.DOT.GOV';
> 'mgoldade@peakpeak.com'

    Cc: Autio, Armi
    Subject: FW: LB-000015, LB-000016, and LB-000017

> For your review and approval.
> - Richard Beckham
> Richard,
> These should be final. The only recent revision is the addition of the
> Effective Date. These need to go to Mark and Mary for their final blessing.
```

```
> <<LB-000015(rev 3_31_03).doc>> <<LB-000016 rev. (3_31_03).doc>>
> <LB-000017 rev(3_31_03).doc>>
> R.K. Mahoney
> Senior Analyst
> Special Projects Coordinator
> EMSL Analytical, Inc.
> Westmont, NJ
> 800.220.3675, x1218
> rmahoney@emsl.com
> << File: LB-000015(rev 3_31_03).doc >> << File: LB-000016 rev. (3_31_03).doc >> << File: LB-000017 rev(3_31_03).doc >> << Fil
```

# Mahoney, Ron

From:

Sent: To:

Subject:

Raney, Mark [RANEY@VOLPE.DOT.GOV] Wednesday, April 23, 2003 9:02 AM 'Mehoney, Ron' FW: EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017





1 9-000018 mm

Ron,

I aimost forgot to forward you this....

See Mary's earlier email below, regarding EPA's approval for MODs L8-15, 16, & 17.

Let me know if you have any questions.

——Original Message——
From: Goldade.Mary@epamail.apa.gov [malito:Goldade.Mary@epamail.epa.gov]
Sent: Thursday, April 03, 2003 5:49 PM
To: Beckham, Richard
Cc: Aurilo, Anni, 'mgoldade@peakpeak.com'; 'RANEY@VOLPE.DOT.GOV'
Subject EPA APPROVED CONDITIONAL: L8-000015, L8-000016, and L8-000017

Mark will modify LB-000015, 16 & 17 to indicate that the Effective Date is: Historical.

"Beckham,

Richard To: <BeckhamRE@cdm.co

EPA approves these mods with this changed completed.

<mgoldade@peakpeak.com>

m>

cc: "Autio, Anni" <AutioAH@cdm.com> Subject: FW: LB-000015, LB-000016, and LB-000017

04/01/03 08:47 AM

For your review and approval.

- Richard Beckham

----Original Message-----From: Mahoney, Ron [mailto:Rmahoney@EMSL.com] Sent Monday, March 31, 2003 6:11 PM To: Beckham, Richard

Subject: LB-000015, LB-000018, and LB-000017

#### Richard,

These should be final. The only recent revision is the addition of the Effective Date. These need to go to Mark and Mary for their final blessing.

<<LB-000015(rev 3\_31\_03).doc>> <<LB-000016 rev. (3\_31\_03).doc>>

R.K. Mahoney Senior Analyst Special Projects Coordinator EMSL Analytical, Inc. Westmort, NJ 800.220.3675, x1218 mahoney@emsl.com

(See attached file: LB-000015(rev 3\_31\_03).doc)(See attached file: LB-000016 rev. (3\_31\_03).doc)(See attached file: LB-000017 rev(3\_31\_03).doc)



# Request for Modification

# To Laboratory Activities LB-000019

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: [All TEM Methodologies]

EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies
Requester: R. K. MahoneyTitle: Senior Analyst/Special Projects Coordinato
Company: EMSL Analytical, Inc. Date: 21 January 2003
Description of Modification: <u>Clarification of bench sheet recording format for grid openings in which no countable structures are recorded.</u>
Reason for Modification:
The electronically deliverable spread sheet for TEM analysis developed for the Libby project requires "ND" (None Detected) to be entered for grid openings in which no countable structures are recorded. The ND code has been used on all electronic deliverables for the Libby project. The code "NSD" (No Structure Detected) has been used on hand written bench sheets up until this date. As of 21 January 2003, "ND" will be used on the
bench sheets as well as the electronically deliverables.
Potential Implications of this Modification: There are no potential negative implications resulting from this clarification of terms.
Laboratory Applicability (circle one): All Individual(s) EMSL Analytical, Inc.
Duration of Modification (circle one):
Temporary Date(s):  Analytical Batch ID:
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages
Permanent (Complete Proposed Modification Section) Effective Date: 21 January 2003 Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.
Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):
Technical Review:
Project Review and Approval:
Approved By:
Title: EPA Regional Chamist (USEPS: Mary Goldade)
(Out G. mary Goldade)

Lab Modification Form Revision 5

#### Mahoney, Ron

From: Sent:

Raney, Mark [RANEY@VOLPE.DOT.GOV] Friday, March 07, 2003 2:50 PM

Beckham, Richard', 'Charlie LaCerra', 'rdemalo@emsl.com', 'rmahoney@emsl.com', Autio, Anni; Raney, Mark; 'brattin@syrres.com', 'Goldade.mary@EPAmail.epa.gov', Montera, Jeff RE: MOD L8-000019

Subject:

I find Laboratory Request for Modification # LB-000019 acceptable as written and here by provide Volpe approval to this

Richard, Please make sure MOD ID#s get inserted onto the mod forms themselves (not just the file ID), so you will be able to identify the IDs based upon hardcopy alone. Also, even though this MOD is applicable to an Individual lab, all MODs are to be forwarded to all labs for informational purposes and to give them an opportunity to provide comments. All labs however are REQUIRED to provide comments to only MODs that are applicable to all labs.

Mark Raney Environmental Engineer

US DOT / Volpe Center Environmental Engineering Division, DTS-33 phone: 617-494-2377 cell: 617-694-8223 fax: 617-494-2789 raney@volpe.dot.gov

This MOD impacts only EMSL. For your review and comment

<<LB-000019.doc>> - Richard Beckham

# Mahoney, Ron

Mary Goldade [mgoldade@peakpeak.com]
Friday, March 07, 2003 12:29 PM
Raney, Mark
Jeff G. Montera; rmahoney@emsl.com; Autio, Anni; William Brattin;
Goldade.Mary@epamail.epa.gov
Re: MOD LB-000019 From: Sent: To: Cc: Subject: I agree that this mod form is acceptable, and should be discussed on the next lab call to be certain similar issues are not encountered at other labs. mary
— Original Message ——
From: "Raney, Mark" <RANEY@VOLPE.DOT.GOV>
To: "Goldade, Mary (HOMÉ)" <mgoldade@peakpeak.com>
Sent Friday, March 07, 2003 10:18 AM
Subject FW: MOD LB-000019

........

> > FYI > ——Original Message——
> From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
> Sent Thursday, March 06, 2003 9:54 AM
> To: 'Charlie LaCerra', 'rdemalo@emsl.com'; 'rmahoney@emsl.com'; Autio,
> Anni; 'Raney@volpe.dot.gov'; 'brattin@syrres.com';
> 'Goldade.mary@EPAmail.epa.gov'; Montera, Jeff
> Subject: MOD LB-000019
> > This MOD impacts only EMSL. For your review and comment > > <<LB-000019.doc>>
> - Richard Beckham



# **Request for Modification**

# To Laboratory Activities LB-000028

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: _	R. K. Mahoney	Title:	Senior Analyst / Special Pr	rojects Coordinator
Company:	EMSL Analytical, Inc.	Date:	17 June 2003	
	of Modification: s a clarification pertaining to the re-	analysis (	of TEM samples when some	of the originally read grid
openings in a	sample selected for re-analysis ha	ve becor	ne unreadable. In the event	that more than half of the
	d grid openings have become unrea ery group with adequate intact grid o			
	e selected are unreadable, make no			
which grid or	penings are unreadable, and procee	d with an	alysis of the original sample.	
Reason for M	Modification: clarification is intended to provide m	ore comr	olete TEM re-analysis data.	
	lications of this Modification:			
There	are no negative implications to this	clarificat	tion.	· · · · · · · · · · · · · · · · · · ·
		/idual(s)		
	lodification (circle one):			
remp	orary Date(s):			
Temporary Mo	dification Forms Attach legible copies	of appro	ved form w/ all associated raw of	data packages
Perm	anent (Complete Proposed Modi	fication S	Section) Effective Date:	17June 2003
Permanent Mo	dification Forms – Maintain legible cop	ies of app	roved form in a binder that can	be accessed by analysts.
Proposed Mo Method when			•	
Technical Re	view: R. K. Washerne (Laboratory Manager or des	onate)	CM5L	Date: 18 July 2003
	w and Approval (Stolpe: Project To	echnical	EMSC  Mark Range  Lead or designate	Date: 7/15/03
Approved By:	: ulaly Goldade			
Title:	Project Chemist or design	ate)		
,	(= === : : , , , , , , , , ,			

Mary Goldade 06/24/03 01:20 PM To: "Beckham, Richard" < BeckhamRE@cdm.com > cc: "Autio, Anni" < AutioAH@cdm.com >, 'Bill Egeland' <br/>
<br/>begeland@mastest.com>, "'Bob.Shumate@battaenv.com'" < Bob.Shumate@battaenv.com > , "'brattin@syrres.com'" <brattin@syrres.com>, 'Charlie LaCerra' <clacerra@emsl.com>, "'corbin77@atc-enviro.com'" < corbin77@atc-enviro.com >, "'dmazzaferro@mastest.com'" <dmazzaferro@mastest.com>, 'Gustavo Delgado' <gdelgado77@atc-enviro.com>, "'Garth B. Freeman'" < gfreeman@mastest.com>, "'jeanneorr@resienv.com'" <jeannearr@resienv.com>, "'mgoldade@peakpeak.com'" <mgoldade@peakpeak.com>, "'m\_szynskie@resienv.com'" <m\_szynskie@resienv.com>, "'Naresh C. Batta'" <ncbatta@battaenv.com>, "'Raney@volpe.dot.gov'" < Raney@volpe.dot.gov>, "'rdemalo@emst.com' <rdemalo@emsl.com>, "'rhatfield@mastest.com'" chatfield@mastest.com>, "'mahoney@emsl.com'" <rmahoney@emsi.com>, 'Shu-Chun Su' <scsu@delanet.com>, 'William Longo' <wlongo@mastest.com>

Subject: Re: EPA Approved w/ revisions MOD LB-000028

EPA approves Mod LB-000028 with revisions as attached.



LB-000028 (MG 6-24-03).

# Mary Goldade

# Regional Superfund Chemist

U.S. Environmental Protection Agency, Region 8 999 19th Street, Suite 300 Mail Code: BEPR-PS

Denver, CO 80202

Fox: (303) 312-6065 email: goldade.mary@epa.gov

Phone: (303) 312-7024

"Beckham, Richard" < BeckhamRE@cdm.com>



"Beckham, Richard" < BeckhamRE@cdm.co m>

06/23/03 08:42 AM

To: 'Charlie LaCerra' < clacerra@ernsl.com > , 'Charlie LaCerra' <clacerra@emsl.com>, "'jeanneorr@resienv.com'" <jeanneorr@resienv.com>, "'rdemalo@emsl.com'" <rdemalo@emsl.com>, "'rmahoney@emsl.com'" <mahoney@emsl.com>, 'William Longo' <wlongo@mastest.com>, "'rhatfield@mastest.com'" <rhatfield@mastest.com>, 'Bill Egeland' <begeland@mastest.com>, "'Bob.Shumate@battaenv.com'" <Bob.Shumate@battaenv.com>, "'Naresh C. Batta'" <ncbatta@battaenv.com>, 'Shu-Chun Su' <scsu@delanet.com>, "'corbin77@atc-enviro.com'" < corbin77@ato-enviro.com > , 'Gustavo Delgado' < gdelgado77@atc-enviro.com>, "'Garth B. Freeman'" <gfreeman@mastest.com>, "Autio, Anni" <AutioAH@cdm.com>, "'Raney@volpe.dot.gov'" <Raney@volpe.dot.gov>, "'brattin@syrres.com'" <brattin@syrres.com>, Mary Goldade/EPR/R8/USEPA/US@EPA, "'dmazzaferro@mastest.com'" <dmazzaferro@mastest.com>,

"'mgoldade@peakpeak.com'" <mgoldade@peakpeak.com>,

"m\_szynskie@resienv.com" <m\_szynskie@resienv.com>

Subject: MOD LB-000028

This MOD impacts all labs. For your review and comment.

- Richard Beckham

<<LB-000028.doc>>

From:

"LaCerra, Charles" <CLaCerra@EMSL.com>

To:

"Carr, Kim" <KCarr@EMSL.com>; "EMSL Mobile Lab - Asbestos" <mobileasbestoslab@EMSL.com>

Sent:

Friday, July 18, 2003 5:57 AM

Attach:

LB-000025\_rev (MG 6-04-03 email).doc; LB-000027 (MG 6-24-03).doc; LB-000028 (MG 6-24-

Sublect:

FW: MODs: LB-000025, 26, 27 & 28

----Original Message-----

From: Raney, Mark [mailto:RANEY@VOLPE.DOT.GOV]

Sent: Friday, July 18, 2003 7:53 AM To: 'Beckham, Richard'; Autio, Anni

Cc: 'Goldade, Mary'; 'Goldade, Mary (HOME)'; 'Orr, Jeaane at Reservoir

Env'; 'Mahoney, Ron'; 'Demalo, Rob (EMSL)'; 'LaCerra, Charles'

Subject: MODs: LB-000025, 26, 27 & 28

Richard.

LB-000025 (EMSL): Volpe provided approval (with revisions) on 6/18/03 & EPA approved on 5/14/03 (see emails and attachment below). I have yet to see a final version for signature. EMSL should finalize, sign and distribute for signature.

LB-000026 (EMSL): Approved and signed by both Volpe and EPA.

LB-000027 (RESI): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). RESI should finalize, sign and distribute for signature.

LB-000028 (EMSL): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). EMSL should finalize, sign and distribute for signature.

Please let me know if anyone has any questions.

Mark.

---Original Message----

From: Beckham, Richard [mailto:BeckhamRE@cdm.com]

Sent: Wednesday, July 16, 2003 5:30 PM To: 'RANEY@VOLPE.DOT.GOV'; Autio, Anni

Subject: MOD Status

For MODs 27 and 28, I have email approvals from EPA, but have not been able

to locate approvals from Volpe. CDM received a hardcopy of 27 with an original signature from RESI, that was subsequently forwarded to Volpe on

7/8/3. (Did I miss an approval email?) To my knowledge, a hardcopy of

has not been prepared.

- Richard Beckham

---Original Message----From: Raney, Mark

Sent: Wednesday, June 18, 2003 10:56 AM

To: 'Mahoney, Ron'

Cc: 'Anni Autio'; 'Mary Goldade'

Subject: RE: EPA Markups: MOD LB-000025

Ron,

I concur with Mary's comments below. I provide Volpe's approval for MOD LB-000025 with Mary's changes and the addition of an estimate of the number of samples involved (i.e., < 20).

Thanks,

Mark.

---Original Message-----

From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]

Sent: Wednesday, June 04, 2003 9:27 AM

To: 'Mark Raney'

Cc: 'Anni Autio'; 'Mary Goldade'; CDM STAFF Subject: FW: EPA Markups: MOD LB-000025

Mark,

Do you have any other comments for this mod? Mary asked for an estimate of the number of samples involved, and we agreed on < 20. The number is more likely < 10, but we've deceided to err on the conservative side.

If I can get your input, we can put this one to bed.

R.K. Mahoney
Senior Analyst
Special Projects Coordinator
EMSL Analytical, Inc.
Westmont, NJ
800.220.3675, x1218
mahoney@emsl.com

----Original Message—
From: Mary Goldade [mailto:mgoldade@peakpeak.com]
Sent: Wednesday, May 14, 2003 6:32 PM
To: Beckham, Richard; 'Charlie LaCerra'; jeanneorr@resienv.com;
rdemalo@emsl.com; rmahoney@emsl.com; 'William Longo';
rhatfield@mastest.com; 'Bill Egeland'; Bob.Shumate@battaenv.com; 'Naresh
C. Batta'; 'Shu-Chun Su'; corbin77@atc-enviro.com; 'Gustavo Delgado';
'Garth B. Freeman'; Autio, Anni; Raney@volpe.dot.gov;
brattin@syrres.com; Goldade.mary@EPAmail.epa.gov;
dmazzaferro@mastest.com; m szynskie@resienv.com
Subject: EPA Markups: MOD LB-000025

Suggested changes to the MOD are attached.
Ron-Do you already have in hand an estimate regarding the actual number of samples this affects (i.e., are you able to quantify the term "few/limited"?)
Thanks,
Mary

Mary Goldade

07/29/03 01:57 PM

To: Anni Autio co: Mark Raney

CC:

Subject: LB-000027 & LB-000028 are signed and mailed

Anni & Joe,

I have mail you the original copiew of the mods LB-000027 & LB-000028. Several of the small approval pages were not provided. I attached them.

# Mary Goldade

# Regional Superfund Chemist

U.S. Environmental Protection Agency, Region 8
999 19th Street, Suite 300
Mail Code: 8EPR-PS
Denver, CO 80202

Phane: (303) 312-7024 Fax: (303) 312-6065

email: goldade.mary@epa.gov



# Request for Modification

to

# Laboratory Activities LB-000029b

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

	one/those applicable): EPA/600/R-93/116 Other:	TEM-AHERA ASTM D5755	TEM-ISO 10312 PC EPA/540/2-90	M-NIOSH 7400 /005a	NIOSH 9002 SRC-LIBBY-03	
Requester:	Lynn Woodbury		Title:	Technical cor	nsultant	
	Syracuse Research C					
standardize the (QC) samples for	ications to laboratory-bifrequency of analysis ar r TEM analyses of air a ut specific details regar	nd procedures for nd dust. The gen	interpretation of the re teral concepts presente	sults for laborated in this modific	ory-based Quality Control sation may also be used for	
Reason for Mod	lification:		•			
					es are prepared in different	
iagoratories in th	e program, and to ensu	ire that all results	are evaluated in accor	d With a standar	o set of criteria.	
	ations of this Modificat lential negative implicat		n this standardization o	of QC procedure	s	
Laboratory Appl	licability (circle one):	All Individu	al(s)	· · · · · · · · · · · · · · · · · · ·		
Temporar	Analytical Bato y Modilication Forms – Atta	ch legible copies of a		•	·	
Permanent (Complete Proposed Modification Section) Effective Date:  Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.						
Data Quality Indicator (circle one) - Please reference definitions on reverse side for direction on selecting data quality indicators:						
Not Appl	icable Reject	Low Blas	Estimate	High Blas	No Bias	
Proposed Modifi when applicable	ication to Method (atta	ch additional she	eets if necessary; stat	e section and p	age numbers of Method	
Technical Revie	w:				Date:	
•	(Laboratory Manag	1 1060 1	100		Date: 4/25/07	
.,	and Approval: (Volpe	Project Technical	l Lead of designate)	<u>-</u>	- <del>- / / / .</del>	
Approved By:	USEPA Projeci Chemisi	soldacle			Date: 475/07	
l	OULI A. 4 TOJELI OJELINSI	or designate)				

18-680a29b v7.dot

# DATA QUALITY INDICATOR DEFINITIONS

**Reject** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

**Estimate** - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

**High Bias** - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

**No Blas** - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

# QC Sample Type Definitions

There are three categories of TEM laboratory QC samples: Blanks, Recounts, and Repreparations.

# Blanks

Lab Blank (LB) – This is a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples.

# **Recounts**

Recount Same (RS) – This is a TEM grid that is re-examined within the same laboratory and by the same microscopist who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Same TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Recount Different (RD) – This is a TEM grid that is re-examined within the same laboratory but by a different microscopist than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Different TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Interlab (IL) - This is a TEM grid that is re-examined by a microscopist from a different laboratory than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Interlab TEM analyses for air and dust will be selected in accord with the procedure presented in Attachment 2.

Verified Analysis (VA) – This is a recount of a TEM grid (same grid openings) performed in accord with the protocol for verified analysis as provided in NIST (1994) (provided as Attachment 3). Verified TEM analyses will be selected in accord with the procedure presented in Attachment 1.

# Repreparations

Repreparation (RP) – This is a TEM grid that is prepared from a new portion of the same filter that was used to prepare the original grid. Typically this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter. Repreparations will be selected in accord with the procedure presented in Attachment 1.

#### Frequency

The minimum frequency for laboratory-based QC samples for TEM analyses (all media combined) shall be as follows:

QC Sample Type	Min. Frequency
Lab blank	4%
Recount same	1%
Recount different	2.5%
Verified analysis	1%
Repreparation	1%
Interlab	0.5%
Total	10%

LB-000029b v7.doc

Each laboratory should prepare and analyze lab blank, recount (same, different and verified), and repreparation samples at the minimum frequency specified in the table above. The selection procedure and laboratory SOP for the selection of samples for the purposes of recounts and repreparation are provided in Attachment 1. Samples for interlab comparisons will be selected by EPA's technical consultant (SRC) in accord with the selection procedure and laboratory SOP provided in Attachment 2.

# Procedure for Evaluating QC Samples and Responses to Exceptions

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

<u>Note</u>: The procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the site to date. These procedures and rules for interpretation may be revised as more data are collected.

#### Lab Blanks.

There shall be no asbestos structure of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples may begin.

## Recounts.

All recount samples (same, different, verified, and interlab) will be evaluated by comparing the raw data sheets prepared by each analyst. Note that the raw data for samples must include sketches for both the initial and QC reanalysis, as described in modification LB-000030. All structure enumeration and measurements will adhere to the established project-specific documentation presented in LB-000016A and LB-000031A. The following criteria will be used to identify cases where results for LA structures are concordant (in agreement) or discordant (not in agreement). These LA criteria were established by microscopists experienced in the analysis of Libby amphibole asbestos, and serve as an initial attempt at review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria for LA structures will be accompanied by scientific justification to support the change. Criteria for concordance on non-LA fibers (OA and C) fibers are the same as described in NIST (1994) (provided as Attachment 3).

Measurement parameter	Concordance Rule
Number of LA asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10%.
Asbestos class of structure (LA, OA, C)	Must agree 100% on chrysotile vs. amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
LA Structure length	For fibers and bundles, must agree within 0.5 um or 10% (whichever is less stringent)
	For clusters and matrices, must agree within 1 um or 20% (whichever is less stringent)
LA Structure width	For fibers and bundles, must agree within 0.5 um or 20% (whichever is less stringent).
	For clusters and matrices, there is no quantitative rule for concordance.

Whenever a recount occurs in which there is one or more discordance, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Whichever analytical result is determined to be correct will be identified with the word "Confirmed" in the sample comment field of the electronic data reporting sheet. In the special case where the original and the reanalysis are both determined to have one or more areas of discordance, a third electronic data report will be prepared that contains the correct results. This will be identified as QA Type = "Reconciliation". The laboratory should maintain records of all cases of discordant results and of actions taken to address any problems, in accord with the usual procedures and requirements of NVLAP. In addition, each laboratory should notify the CDM Laboratory Manager of any significant exceptions and corrective actions through a job-specific (temporary) modification form. The CDM Laboratory Manager will ensure that appropriate Volpe and EPA representatives are notified accordingly.

# Repreparations.

Repreparation samples will be evaluated by comparing the total counts for the original and the re-preparation samples. In order to be ranked as concordant, the results must not be statistically different from each other at the 90% confidence interval, tested using the statistical procedure documented in Attachment 4. Whenever an exception is identified, a senior analyst shall determine the basis of the discordant results, and if it is judged to be related to laboratory procedures (as opposed to unavoidable variability in the sample), the laboratory shall then take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of types, etc).

# Program-Wide Goals

While each laboratory shall monitor the results of the QC samples analyzed within their laboratory and shall take actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. The program-wide goals shall be interpreted as follows:

QC Sample	ample Metric		Program-Wide Criteria		
Type	Wietric	Good	Acceptable	Poor	
Lab Blanks	% with ≥1 asbestos structures	0% - 0.1%	0.2% - 0.5%	>0.5%	
Recounts	Concordance on LA count	>95%	85-95%	<85%	
	Concordance on type (chrysotile vs. amphibole)	>99%	95%-99%	<95%	
	Concordance on LA length	>90%	80%-90%	`<80%	
	Concordance on LA width	· >90%	80%-90%	<80%	
Repreps	Concordance on LA concentration/loading	>95%	90-95%	<90%	

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

#### REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

# **ATTACHMENT 1**

Selection Procedure and Laboratory SOP for Recounts (RS, RD, VA) and Repreparations (RP)

# Selection Procedure

As specified in the Frequency section above, the frequency of Recount Same (RS) should be 1%, the frequency of Recount Different (RD) should be 2.5%, the frequency of Verified Analyses (VA) should be 1%, and the frequency of Repreparations (RP) should be 1%, corresponding to a total within-laboratory QC frequency of 5.5% for these analysis types. This is approximately 1 QC sample per 20 field samples. Based on this frequency, it is possible to determine which laboratory job(s) will have one or more samples selected for recount analysis or repreparation.

For those laboratory jobs in which a recount or repreparation sample is to be selected, the analyst should record the total number of structures observed in each sample. The sample(s) selected for recount or repreparation should be those within the laboratory job with the highest number of structures per grid opening (GO) area examined (calculated as the number of GOs evaluated \* the GO area). When selecting samples for repreparation, if possible, preferentially select samples in which the total number of GOs is 40 or less. Because repreparations concordance is evaluated based on concentration, in order to achieve adequate statistical power, repreparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the selection of samples with 40 GOs or less will reduce analytical costs associated with repreparations. When selecting samples for recount, it is not necessary to impose a minimum or maximum number of GOs because concordance is evaluated on a GO and structure basis, rather than a concentration basis. If all samples within the laboratory job are non-detect, a non-detect sample may be selected. A non-detect sample should be preferentially selected, every 10<sup>th</sup> selection.

This selection procedure will ensure that the recount analyses and repreparations yield a dataset best suited to assess concordance<sup>1</sup>.

# Laboratory SOP for Recount Analyses

- 1. For recount samples, re-analyze the selected sample in accord with the appropriate procedures for each type of recount (RS, RD, or VA). If more than 10 GOs were evaluated in the original analysis, the original analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the recount analysis. The original analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures per GO area examined, which may be analyzed in the event that a selected GO is damaged and cannot be re-evaluated.
- 2. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Recount Same", "Recount Different", or "Verified Analysis", as appropriate. Be sure that the grid and GO names match exactly with the names evaluated in the original analysis (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the original analyst or laboratory director to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).
- 3. If there is one or more discordant GOs between the original analysis and the recount analysis, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

<sup>&</sup>lt;sup>1</sup> It should be noted that this selection procedure will tend to result in the preferential selection of samples with the highest air concentration/dust loading values. Thus, summary statistics based on laboratory QC samples may tend to be biased high.

LB-000029b y7.doc

4. Submit the recount TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

# Laboratory SOP for Repreparations

- 1. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.
- Select two grids and read the same number of total GOs as the original analysis, using the TEM counting
  rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original
  analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation.
  Place the remaining grid in storage.
- 3. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".
- 4. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

#### **ATTACHMENT 2**

# Selection Procedure and Laboratory SOP for Interlabs (IL)

# Selection Procedure

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- 1. On the 1st of each month, EPA's technical consultant (SRC) will compile a list of all samples for which air and dust TEM results (ISO+AHERA+ASTM) were uploaded into Libby V2 Database in the preceding month (e.g., on November 1<sup>st</sup>, specify a date range of Oct 1-31, 2005). The Libby V2 Database query will be based on the upload date rather than the analysis date to ensure that analyses with an upload in a different month as the analysis date were not excluded<sup>2</sup>.
- 2. Identify the target number of air and dust interlab samples needed to meet the QC requirements for interlabs specified in the Frequency section above (0.5%). This is accomplished by multiplying the desired interlab frequency (0.5%) by the total number of air and dust analyses performed in the preceding month. For example, 178 TEM air analyses in October 2005 \* 0.5% = 0.89 (which is rounded up to 1). At a minimum, at least one air and one dust sample will be selected for interlab analysis.
- 3. For each medium (air and dust), rank order the TEM analyses from the preceding month on the total number of LA structures per GO area examined (calculated as the number of GOs evaluated \* the GO area). Selecting from analyses with a high number of LA structures per GO area examined increases the likelihood that the GOs evaluated as part of the interlab analysis will have one or more LA structures.
- 4. Exclude samples in which the total number of GOs is more than 40 GOs<sup>3</sup>. Exclude any samples that have already been selected for interlab evaluation previously.
- 5. Select the appropriate number of air and dust interlab samples from the available TEM analyses for which the total number of LA structures per GO area examined is higher than 0 (i.e., LA detects). If the total number of samples with LA detects is equal to the desired number of interlab samples, select all detected samples for interlab analysis. If the total number of samples with LA detects is less than to the desired number of interlab samples, select non-detect samples for interlab analysis. If the total number of samples with LA detects is higher to the desired number of samples, interlab samples will be selected to represent multiple laboratories, selecting those samples with the highest number of LA structures per GO examined first. EPA's technical consultant (SRC) will keep a running total of the number of samples selected by laboratory to ensure that the long-term frequency of interlabs for each laboratory is generally similar.
- 6. Submit list of selected interlab samples to the CDM Laboratory Manager.
- 7. Each month, the CDM Laboratory Manager will provide each laboratory with the list of samples selected for Interlab analysis.

<sup>&</sup>lt;sup>2</sup> Consider the case where the TEM analysis for sample X-12345 was performed on September 22 and the results were uploaded on October 3. The interlab selection query performed on October 1, if limited to all results analyzed from September 1-30, would not capture the results for X-12345 because they had not yet been uploaded. The interlab selection query performed on November 1, limited to all results analyzed from October 1-31, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

<sup>&</sup>lt;sup>3</sup> Because all interlabs will be reprepared, these interlab repreparation samples will also be evaluated for concordance with the original sample. Because repreparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, repreparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the focusing on samples with 40 GOs or less will reduce analytical costs associated with repreparations.

# **Laboratory SOP**

# At the Originating Laboratory:

- 1. Upon receipt of the interlab sample list from the CDM Laboratory Manager, locate the appropriate sample filter. If less than ¼ of the sample filter is available, contact the CDM Laboratory Manager to identify an interlab replacement sample.
- 2. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.
- 3. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.
- 4. Record the orientation of each grid using the instructions for grid orientation specified in NVLAP (see Attachment 5).
- 5. When performing the TEM analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.
- 6. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".
- 7. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.
- 8. Identify which laboratory will perform the interlab analysis in accord with the following table:

Originating Lab	Lab for interlab Sample #1	Lab for Interlab Sample #2	Lab for Interlab Sample #3	Lab for Interlab Sample #4	Lab for Interlab Sample #5	Lab for Interlab Sample #6
Hygeia	Batta	MAS	RESI	EMSL-L	EMSL-W	Danast
Batta	MAS	RESI	EMSL-L	EMSL-W	Hygeia	Repeat
MAS	RESI	EMSL-L	EMSL-W	Hygeia	Batta	(beginning with the Lab
RESI	EMSL-L	EMSL-W	Hygeia	Batta	MAS	identified for
EMSL-L	EMSL-W	Hygeia	Batta	MAS	RESI	Sample #1)
EMSL-W	Hygeia	Batta	MAS	RESI	EMSL-L	Carriple #17

EMSL-L = EMSL, Mobile Lab in Libby EMSL-W = EMSL, Westmont

- 9. If more than 10 GOs were evaluated in the repreparation analysis, the repreparation analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the interlab analysis. The repreparation analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures, which may be analyzed in the event that the selected GO is damaged and cannot be re-evaluated.
- 10. Ship the grid(s) for the interlab sample to the appropriate laboratory using standard chain of custody procedures. For each interlab sample, include a list of which GOs should be evaluated for each grid. The names of the grid and GOs provided on the chain of custody form should match exactly with those recorded in the original TEM data recording spreadsheet (including dashes, underscores, and spaces).
- 11. After the interlab laboratory has completed the interlab analysis, it will request copies of the hard copy laboratory benchsheet(s), the grid opening sketches, and TEM file for each interlab sample.

12. If areas of discordance are noted, the senior laboratory analyst from the interlab laboratory will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

# At the Interlab Laboratory:

- 1. For each grid provided for interlab analysis, place the grid into the TEM grid holder ensuring that the grid orientation matches that which was specified by the originating laboratory (see Attachment 5 for details).
- 2. For the 10 GOs identified for interlab analysis, perform TEM analysis using the analysis method and counting rules specified on the chain of custody. Be sure that the grid and GO names match exactly with the names provided on the chain of custody (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), <u>DO NOT</u> arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the originating laboratory to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).
- 3. When performing the TEM interlab analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.
- 4. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Interlab".
- 5. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.
- 6. Contact the originating laboratory to request copies of the hard copy laboratory benchsheet(s), grid opening sketches, and TEM file for each interlab sample.
- 7. Perform a verified analysis using the procedures presented in NIST (1994) (provided as Attachment 3).
- 8. Assess the between-laboratory concordance, both on a GO-by-GO basis and on a structure-by-structure basis, using the Libby-specific recount concordance rules. If areas of discordance are noted, the senior laboratory analyst will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).
- Summarize the results of the verified analysis and document any changes in laboratory procedures or analyst training that were implemented to address noted discordances. Provide a copy of this report to EPA Chemist and the CDM Laboratory Manager.
- 10. Ship the grid(s) back to the originating lab.

# **ATTACHMENT 3**

Airborne Asbestos Method:
Standard Test Method for Verified Analysis of Asbestos
by Transmission Electron Microscopy-Version 2.0.
NIST (1994)

# NISTIR 5351

Alrborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy -Version 2.0

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TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY Arati Prabhakar, Director

# Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

## 1. Scope

- I.I This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.
- 1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.
- 1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).
- 1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysts use the same counting rules.

# 2. Terminology

- 2.1 Definitions:
- 2.1.1 TEM--transmission electron microscope.
- 2.1.2 grid square, grid opening--an area on a grid used for analysis of asbestös by transmission electron microscopy.
- 2.1.3 verified analysis—a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification is recorded for each observed structure.
- 2.1.3.1 Discussion—Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.
  - 2.2 Description of Terms Specific to This Standard:
- 2.2.1 counting rules—rules used to determine the amount of asbestos present in an asbestos-containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).
- 2.2.2 AHERA method'--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.
- 2.2.3 ISO method<sup>2</sup>--procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.
  - 2.2.4 particle—an isolated collection of material deposited on a grid or filter.
- 2.2.5 structure—a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.
- 2.2.6 TEM operator, TEM analyst-person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.
- 2.2.7 verifying analyst--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

<sup>&#</sup>x27;Code Fed, Reg. 1987, 52 (No. 210), 41826-41905.

<sup>&</sup>lt;sup>2</sup>ISO 10312 1993, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

- 2.2.8 TEM analysis form—form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.
- 2.2.9 report form—form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.
  - 2.2.10 SR (structures reported)—the number of structures reported by a TEM analyst.
- 2.2.11 *TP (true positive)*--structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.
- 2.2.12 TPM (true positive-matched)—structure that is reported on the TEM analysis forms of both TEM operators.
- 2.2.12.1 Discussion.—To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.
- 2.2.13 TPU (true postive-unmatched)--structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.
- 2.2.14 TPV (true positive found by verifying analyst) -- structure not found by the two TEM operators but found by the verifying analyst.
- 2.2.15 TNS (total number of structures)—the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.
- 2.2.15.1 Discussion—The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.
- 2.2.16 FN (false negative)—structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories-type A and type B as discussed in the next two terms.
- 2.2.17 FNA (false negative-type A)—false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.
- 2.2.18 FNB (false negative-type B)--false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.
- 2.2.19 FP (false positive)—reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.
  - 2.2.20 TN (true negative)—reported particle that is correctly characterized as zero structures.
- 2.2.21 NL (not located structure)—structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.
- 2.2.21.1 Discussion—The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.
- 2.2.22 AMB (ambiguous structure)—a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.

# 3. Significance and Use

- 3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analysts participating in the verified count.
- 3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/INS and FP/INS can be plotted vs time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/INS values  $\geq 0.85$  and FP/INS values  $\leq 0.05$ . The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.
- 3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

# 4. Procedure

NOTE I-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

- 4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.
- 4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.
- NOTE 2— The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.
- 4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification for instance, nearby particles, sample preparation features or grid bars.
  - 4.2 Submit the analyses of the two TEM operators to the verifying analyst.
- NOTE 3— The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.
- 4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.
- NOTE 4— The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

- 4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight foward as shown in Fig. X2.2 X2.4 of Appendix X2 or more complex as described in the next item.
- 4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.
- 4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.
- NOTE 5— The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.
- 4.5.1 Determine if the reported structure can be located. If the structure cannnot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).
- 4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.
- 4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.
- 4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).
  - 4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).
- 4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator \_ did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.
- 4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analysis TEM analysis form.
- 4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled

TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6-4.5.7.

4.7 Complete the report form as described in items 4.7.1-4.7.10.

- 4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.
- 4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.
- 4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.
- 4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).
  - 4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.
- 4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).
  - 4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.
- 4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.
- 4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.
  - 4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).
  - 4.8 Check the numbers on the report form using the equations given in the calculation section.

#### 5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$I = TP/TNS + FN/TNS$$

6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6-- The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

#### 7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

Grid box: \_\_\_\_

## **APPENDIXES**

## (Nonmandatory Information)

## X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box:		Date:				
Grid slot:		Verifying Analyst:				
Grid square:						
		-				
	Analysis l	Analysis 2				
TEM Operator						
Structures Reported (SR)						
True Positives (TP)						
*TPM						
TPU						
*TPV						
*Total # Structures (TNS)						
False Positives (FP)						
False Negatives (FN)						
FNA						
FNB						
Not Located (NL)						
Ambiguous (AMB)						
TP/TNS						
FP/TNS						

<sup>\*</sup>The values for these items will be the same for both analyses.

Test Report Form (continued)

1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

## X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

## Analyst 1

## Length (µm) Verification Structures Width (µm) .Sketch ₽ 1.3 0.1 TPM<sub>1</sub> 1 Chr 1 Chr 0.7 0.1 TPM2 1.0 0.1 TPM3 1 Chr

Length (µm)	Width (pm)	-Sketch	Verification	# Structures	₽
1.3	0.1		ТРМ1	1	Chr
1.0	0.1		ТРМ3	1	Chr
0.7	0.1	1/	ТРМ2	1	Chr

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts.

— Matching structures are indicated by TPM(number).

Length (pm)	Width (µm)	Sketch	Verification	# Structures	Ω.	Length (µm)	Width (µm)	Sketch	Verification	# Structures	Q)
1.3	0.1		ТРМ1	1	Chr	1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr	1.0	0.1		ТРМ3	1	Chr
1.0	0.1		тРМ3	1	Chr	0.7	0.1	1	TPM2	1	Chr
0.7	0.1	-	FP1	1	Chr						

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, origination and a similar nearby particle). The duplicate structure is therefore assigned an FP1.

Analyst 2

(mr) djoue l	(mid) influen	Width (µm)	Sketch	Verification	# Structures	9
0	.6	0.1	/	TPU1	1	Сћг

Length (µm)	Width (µm)	Sketch	Verification	# Structures	Ω
0.6	0.1		FNA1	0	Сћг

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Length (µm)	Width (µm)	Sketch	Verification	# Structures	Ō
0.4	0.1		FP1	1	Chr

ட்சாழி (பா)	Width (µm)	Sketch	Verlitcation	# Structures	0
0.4	0.1		TN1	0	Chr

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.

Analyst 2

Length (pm)	Width (µm)	Sketch	Verification	# Structures	Ō	Length (µm)	Width (µm)	Sketch	Verification	# Structures	ð
1	0.6		TPM1 FNA1	1	Chr			F1 F2			
						1	0.1	- F1	ТРМ1	1	Chr
						0.6	0.1	. F2	TPU1	1	Chr

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

Length (µm)	Width (µm)	Sketch	Verification	# Skructures	Q	ւեոցլի (µm)	Width (µm)	Sketch	Verification	# Structures	Ō
5	3	<b>*</b>	TPM1	1	Chr			F1 F3			
ž						5	0.1	- F1	ТРМ1	1	Chr
						3	0.1	F2	FP1	1	Chr
			·			2	0.1	F3	FP2	1	Chr
						1	0.1	F4	FP3	1	Chr

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysis have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.

Ō

Chr

₽

Chr

Ö

Chr

C

FP1

1

b

a

0.4

0.1

#### Analyst 1 Analyst 2 Length (புர) Verification Structures Length (µm) Verification Structures Width (pm) Width (µm) Sketch Sketch $\Box$ 0.4 0.1 0 0.6 0.1 1 Chr Structures Length (µm) Structures Length (µm) Verification Verification Width (µm) Width (µm) Sketch Sketch Q 0.4 0.1 0 Chr 0.6 0.1 TPU1 1 FNA1 Structures Structures Length (µm) Verification Verification Length (µm) Width (µm) Width (pm) Sketch Sketch Ω

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 is assigned a TN1 and analyst 2 an FP1.

0

TN1

Chr

0.6

0.1

## Structures Length (um) Width (µm) Verification Sketch ₽ 1.3 0.1 TPM1 1 Chr 0.6 1 0.1 Chr 0.1 1.0 1 TPM2 Chr

## Analyst 2

Length (um)	Width (um)	Sketch	Verification	# Structures	Q
1.3	0.1		TPM1	1	Chr
1.0	0.1		ТРМ2	1	Chr

a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).

C

0.1

1.0

## Analyst 1

## # Structures Length (um) Verification Width (pm) Sketch ₽ 1.3 0.1 TPM1 1 Chr TPU1 1 0.6 0.1 Chr

1

TPM2

Chr

## Analyst 2

Length (um)	Widih (µm)	Sketch	Verification	# Structures	Ō
1.3	0.1		TPM1	1	Chr
1.0	0.1		FNB1 TPM2	1	Chr
		·			

Length (um) # Structures Width (µm) Verification Sketch ₽ 1.3 0.1 TPM1 1 Сһг 1 Chr NL1 0.6 0.1 1.0 0.1 TPM2 1 Chr

Length (um)	Width (µm)	Sketch	Verification	# Structures	Q
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

Fig. X2.8 (caption on previous page).

9

Chr

Chr

Chr

Chr

Structures

1

1

1

1

Verlication

## Analyst 1

## Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	Ō	Length (µm)	Widlh (µm)	Skelch
5	3	X		1	Chr			F1 F3
						5	0.1	F1
						3	0.1	F2
	:					2	0.1	F3
						1	0.1	F4

a

Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).

b

Analyst 1

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	QI	
5	3	<i>X</i>	ТРМ1	1	Chr	
					·	

	1				
Length (µm)	Width (pm)	Sketch	Verification	# Structures	Q
		F1 F3			
5	0.1	F1 -	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

Fig. X2.9 (caption on previous page)

#### X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS

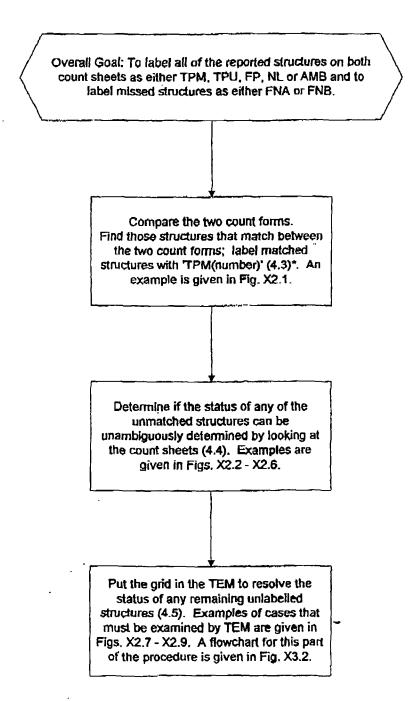


Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst. \*Numbers in parentheses in each block refer to the item number in the procedure.

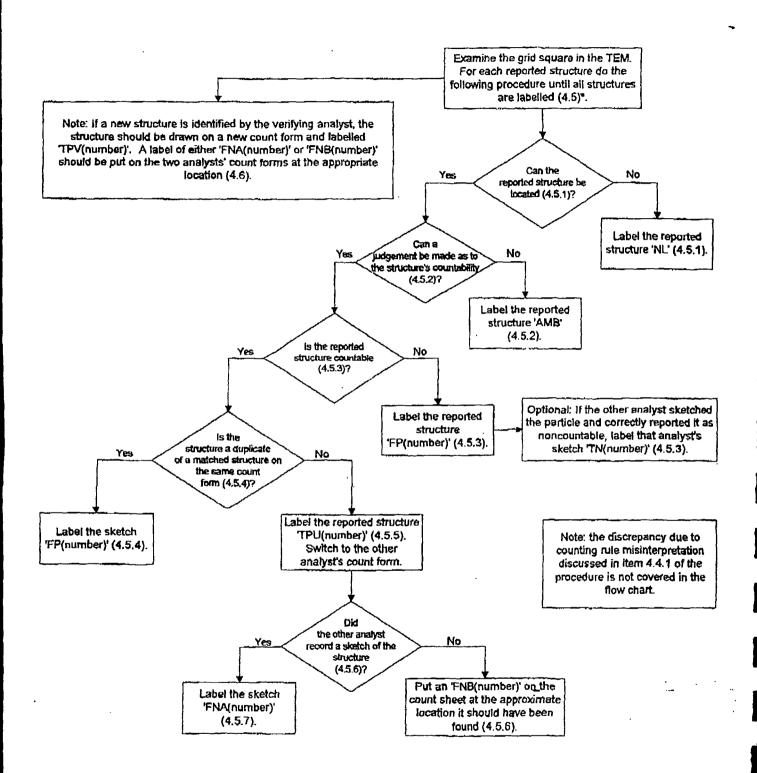


Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. \*Numbers in parentheses in each block refer to the item number in the procedure.

## Statistical Comparison of Two Poisson Rates

#### 1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the repreparation and reanalysis of a number of TEM grids for quantification of asbestos fiber concentrations in air and dust. Because of random variation, it is not expected that results from repreparations samples should be identical. This attachment presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

#### 2.0 STATISTICAL METHOD

This method is taken from "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

N1 = Fiber count in first evaluation

S1 = Sensitivity of first evaluation

N2 = Fiber count in second evaluation

S2 = Sensitivity of second evaluation

The test is based on the confidence interval around the ratio of the two observed Poisson rates:

Rate 1 = N1 · S1

Rate 2 = N2 · S2

Ratio = Rate 1 / Rate 2

Lower Bound = 
$$\left(\frac{S1}{S2}\right)\left(\frac{N1}{N2+1}\right)/F\left[\frac{1+\gamma}{2}; 2\cdot N2+2, 2\cdot N1\right]$$

Upper Bound = 
$$\left(\frac{S1}{S2}\right)\left(\frac{N1+1}{N2}\right) \cdot F\left[\frac{1+\gamma}{2}; 2 \cdot N1 + 2, 2 \cdot N2\right]$$

where  $\gamma$  is the confidence interval (e.g., 0.95) and F[ $\delta$ ; df1, df2] is the 100 $\delta$ th percentile of the F distribution with df1 degrees of freedom in the numerator and df2 degrees of freedom in the denominator.

If the lower bound of the ratio is > 1, then it concluded that rate 1 is greater than rate 2 at the  $100(1-\gamma)\%$  significance level. If the upper bound of the ratio is < 1, then it concluded that rate 1 is less than rate 2 at the  $100(1-\gamma)\%$  significance level. Otherwise, it is concluded that rate 1 and rate 2 are not different from each other at the  $100(1-\gamma)\%$  significance level.

#### Example:

N1 = 4 structures

 $S1 = 0.0001 (cc)^{-1}$ 

Rate  $1 = 4 \cdot 0.0001 = 0.0004$  s/cc

N2 = 6 structures

 $S2 = 0.001 (cc)^{-1}$ 

Rate  $2 = 6 \cdot 0.001 = 0.006$  s/cc

y = 0.95

Lower Bound = 
$$\left(\frac{0.0001}{0.001}\right)\left(\frac{4}{6+1}\right) / F\left[\frac{1+0.95}{2}; 2\cdot 6+2, 2\cdot 4\right] = 0.014$$
  
Upper Bound =  $\left(\frac{0.0001}{0.001}\right)\left(\frac{4+1}{6}\right) \cdot F\left[\frac{1+0.95}{2}; 2\cdot 4+2, 2\cdot 6\right] = 0.281$ 

In this example, because the upper bound of the ratio is < 1, it is concluded that Rate 1 (0.0004 s/cc) is less than Rate 2 (0.006 s/cc) at the 95% significance level.

## 3.0 REFERENCES

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

## **ATTACHMENT 5**

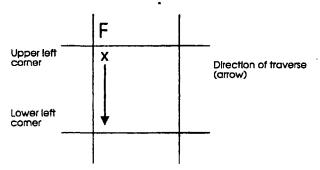
NVLAP Airborne Asbestos Proficiency Test 98-2: Grid Orientation

#### **NVLAP AIRBORNE ASBESTOS PROFICIENCY TEST 98-2**

## Instructions for Form 1

The following procedure is designed to ensure that all laboratories count the grid squares in the same orientation and scan direction to allow for verified analyses which will be performed in the next round of proficiency testing.

- 1. Put a grid into the TEM. Find a particle at the magnification typically used for asbestos analysis. Move the particle using one stage translation and record the direction of movement of the particle on Form 1. Move the particle using the other stage translation knob and record the direction of movement. Recording the two directions of movement should roughly form a cross. The cross represents the translation directions of your microscope at the magnification used for asbestos analysis. Draw the letter "F" onto the cross so the sides of the letter are parallel to the translation directions and the letter is upright and is not inverted. See the example on Form 1.
- 2. Decrease the magnification and locate the letter "F" on the finder grid. Increase the magnification of the TEM to that typically used for asbestos analysis by your lab, keeping the letter "F" in the field of view. Compare the orientation of the "F" to the cross drawn in step 1. If the letter "F" is not oriented as shown in your sketch, remove the specimen holder and rotate or invert the grid as necessary to correctly align the grid. This may require several iterations.
- 3. When the correct orientation is found, record the grid's position in the specimen holder as shown in the example of the second part of *Form 1*. Indicate in your drawing where the straight side and the notched portion of the grid are located. All grids analyzed in this proficiency test should be oriented in the same manner (always check that the letter "F" is in the correct orientation and that the X-Y translation directions allow translation roughly parallel to the grid bars).
- 4. The starting point of the traverse for structure counting must correspond to the upper left corner on the grid square. The "X" marks the starting corner of the traverse (your grid square may be at an angle to that shown in the example):



The initial direction of traverse must be from the upper left corner to the lower left corner of the grid square. If correctly oriented, the edge of the grid bar will remain in the field of view during the entire initial traverse (some allowance must be made for curvature or irregularly shaped grid bars.) If the grid is not oriented properly, go back to step 2.

**NVLAP AIRBORNE ASBESTOS PROFICIENCY TEST 98-2** 

•	
NVLAP Lab	Cada
INVLAT Lab	Coue:

## Form 1. Grid Orientation

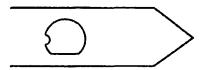
1. Sketch the orientation of the X-Y translation directions of the electron microscope as projected onto the electron microscope stage. Record the letter "F" as shown in the example below:

**EXAMPLE:** 



2. Sketch below the orientation of the grid relative to the sample holder as shown in the example below:

**EXAMPLE**:



## **ATTACHMENT 6**

Grid Opening Template for Sketching the Relative Position of Observed Structures

		Page	_ of
	JRE LOCATIONS WITHIN completed for interlab analyse		
Lab Name:			
Index ID:			
	Reprep for interlab		
Grid:	Grid Opening: _	 	
r			
r		•	
1			

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## **Request for Modification**

# To Laboratory Activities LB-000030

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Lab Applicable forms — copies to: EPA, Volpe, CDM-Denver, All project labs

Individual Lab Applicable forms - copies to: EPA, Volpe, CDM-Denver, Initiating Lab Method (circle one/those applicable): TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2) Requester: W.J. Brattin Title: Technical consultant Company: Syracuse Research Corporation Date: 5 August 2003 Description of Modification: All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of stricture appearance, and orientation relative to any nearby landmarks, if present. Cmorphology, Reason for Modification: This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no loner need to be identified before analysis. Potential Implications of this Modification: There are no potential negative implications resulting from this standardization of QC procedures. Laboratory Applicability (circle one): All Individual: Duration of Modification (circle one): Temporary Date(s): Analytical Batch ID: Temporary Modification Forms - Attach legible copies of approved form w/ all associated raw data packages (complete Proposed Modification Section) Effective Date: (insert based on date of final approval) |Permanent| Permanent Modification Forms - Maintain legible copies of approved form in a binder that can be accessed by analysts. Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): Technical Review: (Laboratory Manager or designate) Project Review and Approval:

(Volpe: Project Technical Lead or designate)

## Autio, Anni

From: Sent:

Goldade.Mary@epamail.epa.gov Thursday, August 07, 2003 10:43 AM

Autio, Anni

To: C¢:

Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos'; ncbatta@battaenv.com; Marie Cash; 'emseque (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield;

Subject:

Ron Mahoney; Shu-Chun Su; Bill Longo EPA Comments: LB-000030 (Draft for review/comment)





LB-000030 v0 (MG pic08313.gif (3 KB) 08-07-03).doc...

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)



## **Request for Modification**

#### To Laboratory Activities LB-000030

instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): [FEM-AHERA, TEM-ISO 10312], PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: EPA/600/R-94/134 (EPA 100.2)

Requester: W.J. Brattin	Title: Technical consultant	
Company: Syracuse Research Corporation	Date: 5 August 2003	
	sketches of all asbestos structures observed, up to a maximum of 50	
	e highly detailed, but should include an indication of structure	Deleted:
appearance, morphology and orientation relative to	any nearby landmarks, if present.	
aspestos structures. One benefit of this modificatio	the procedure used by each laboratory for recording sketches of on is that samples for verified analysis no longer need to be identified to laboratory's supervisor or designate following analysis.	_
that samples selected for verified analyses will be u		
Laboratory Applicability (circle one): All Inc	dividual:	<del>-</del>
Duration of Modification (circle one): Temporary Date(s): Analytical Betch ID:		_
	of approved form w/ atl associated raw data packages	_
Permanent] (complete Proposed Modifi Permanent Modification Forms – Maintain legible copi	ication Section) Effective Date: finaert based on date of final approval) ies of approved form in a binder that can be accessed by analysts.	_
Proposed Modification to Method (attach additional applicable):	sheets if necessary; state section and page numbers of Method when	<u></u>
		Deleted:
Technical Review: (Laboratory Manager or de	.signate) Date:	_
Project Review and Approval: <u>(Volpe: Project Techni</u>	ical Lead or designate)	
Approved By:(USEPA: Project Chemist or design	Date:	_
(USEPA: Project Chemist or design	nate)	

Modification for Lab QC

#### ·Autio, Anni

From: Sent:

DeMalo, Robert [RDemalo@EMSL.com] Thursday, August 07, 2003 11:20 AM

To:

Cc:

Goldade.Mary@epamail.epa.gov; Autio, Anni Bob Shumate; LaCerra, Charles; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr, Kwiatkowski, Joseph; Marie Cash; EMSL Mobile Lab - Asbestos; ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Richard Hatfield; Mahoney,

Subject:

Ron; Shu-Chun Su; Bill Longo RE: EPA Comments: LB-000030 (Draft for review/comment)

I propose adding the word "morphology" as well into the description, as noted. I have no problem with including ISO to this procedure.

----Original Message----

From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]

Sent: Thursday, August 07, 2003 10:43 AM

To: Autio, Anni

Cc: Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth

Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos';

ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron

Mahoney; Shu-Chun Su; Bill Longo

Subject: EPA Comments: LB-000030 (Draft for review/comment)

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.qif)

## Autio, Anni

From: Sent:

Raney, Mark [RANEY@VOLPE.DOT.GOV] Thursday, August 14, 2003 10:41 AM

To: Cc: 'Goldade.Mary@epamail.epa.gov'; Autio, Anni Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth Freeman; Jeanne Orr, Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos';

ncbatta@battaenv.com; Raney, Mark; Rob DeMalo; Richard Hatfield; Ron Mahoney; Shu-

Chun Su; Bill Longo

Subject:

RE: EPA Comments: LB-000030 (Draft for review/comment)



LB-000030 v0 (MR 08-14-03).doc...

I concur with Mary's recommendations and mark-ups. The attached version also includes Rob Demalo's recommendation of adding morphology under the description section. Bill please finalize, sign and send it through the signature process. To expedite the process could you get Mary to sign before providing the original on for my signature. Let me know if you have any questions.

Thanks,

Mark.

----Original Message----

From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]

Sent: Thursday, August 07, 2003 10:43 AM

To: Autio, Anni

Cc: Bob Shumate; Charlie LaCerra; Kyeong Corbin; Denise Mazzaferro; Gustavo Delgado; Garth

Freeman; Jeanne Orr; Kwiatkowski, Joseph; Marie Cash; 'EMSL Mobile Lab - Asbestos';

ncbatta@battaenv.com; Mark Raney (raney@volpe.dot.gov); Rob DeMalo; Richard Hatfield; Ron

Mahoney; Shu-Chun Su; Bill Longo

Subject: EPA Comments: LB-000030 (Draft for review/comment)

Attached are my recommended mark-ups. I also included Jeanne's recommendation of "if present" after landmarks. Please review and comment as nec.

One other point of clarification....when we discussed this, we were focused on AHERA. Just want to make sure it's OK w/ all to include TEM ISO on this list of circled methods. Thanks, Mary (See attached file: LB-000030 v0 (MG 08-07-03).doc) (Embedded image moved to file: pic08313.gif)



## Request for Modification To

#### Laboratory Activities LB-000030

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM), Data Manager distributes approved forms as follows:

All Lab Applicable forms - copies to: EPA, Volpe, CDM-Denver, All project labs Individual Lab Applicable forms - copies to: EPA, Volpe, CDM-Denver, Initiating Lab Method (circle one/those applicable): [TEM-AHERA, TEM-ISO 10312]. PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, [ASTM D5755-95], EPA/540/2-90/005a, Other: [EPA/600/R-94/134 (EPA 100.2)] Requester: W.J. Brattin Title: Technical consultant Company: Syracuse Research Corporation Date: 5 August 2003 Description of Modification: All samples analyzed by TEM shall include sketches of all asbestos structures observed, up to a maximum of 50 structures in a sample. These sketches need not be highly detailed, but should include an indication of structure Deleted: I appearance, morphology and orientation relative to any nearby landmarks, if present. Reason for Modification: This modification is needed to standardize the procedure used by each laboratory for recording sketches of asbestos structures. One benefit of this modification is that samples for verified analysis no longer need to be identified before analysis and will be randomly selected by the laboratory's supervisor or designate following analysis. Potential Implications of this Modification: There are no potential negative implications resulting from this standardization of QC procedures, but a benefit is that samples selected for verified analyses will be unknown to the microscopist prior to analysis. Laboratory Applicability (circle one): Individual: Duration of Modification (circle one): Date(s): Temporary Analytical Batch ID: Temporary Modification Forms - Attach legible copies of approved form w/ all associated raw data packages Permanent | (complete Proposed Modification Section) | Effective Date: (insert based on date of final approval)
Permanent Modification Forms - Maintain legible copies of approved form in a binder that can be accessed by analysts. Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): Technical Review: \_\_ Date: (Laboratory Manager or designate) Project Review and Approval: Date: (Volpe: Project Technical Lead or designate)

Date:

Modification for Lab QC

Approved By:\_

(USEPA: Project Chemist or designate)



## **Request for Modification**

# to Laboratory Activities LB-00066c

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle	one/those applicable): EPA/600/R-93/116 Other:	TEM-AHERA ASTM D5755	<del></del>	② PCM-NIOS⊢ 0/2-90/005a -	17400 NIOSH 9002 SRC-LIBBY-03
Requester:	W. Brattin		Title: _	Technical Cons	ultant
Company:	Syracuse Research Co	orporation	Date: _	09/11/2007	

#### Description of Modification:

This temporary modification applies to all investigative samples (as defined by the most recent version of LB-000053) evaluated at the Libby Superfund site. Based on this temporary modification, all analytical laboratories shall: 1) begin to utilize the structure comment field to further characterize particles with regard to the levels (presence/absence) of the sodium and potassium peaks observed in the EDS spectrum; 2) record on the data sheets all NAM particles that are "close calls" (defined in attachment 1); 3) increase the frequency that EDS spectra are saved for "LA" and "close call" structures; 4) increase the frequency that photographic images of particle morphology are recorded for "LA" and "close call" structures, and 5) utilize the comment field to record mineral type of each recorded particle, including LA, OA, C and "close call" NAM particles,

#### Reason for Modification:

Studies of asbestos from the mine in Libby indicate that the asbestos spans several different mineralogical classes, including winchite and richterite (these are the primary forms) as well as tremolite and possibly actinolite (these are minor forms) (Meeker et al, 2003). Consequently, all analytical laboratories supporting the Libby project are currently directed to classify as "LA" any particle in an investigative sample that a) meets morphological requirements (e.g., length ≥ 0.5 um, aspect ratio ≥ 3:1), b) has an SAED diffraction pattern that is consistent with amphibole, and c) has an EDS spectrum that is consistent with the range of mineral forms observed in the mine in Libby (USEPA 2005). To date, this method for designating "LA" to a particle has worked well for samples collected at the Libby Site. However, a recent project that included collection of air samples from locations outside of Libby highlighted a potential limitation of this approach. That is, tremolite and actinolite are included in the "LA" suite and are found in Libby, but these types of fibers may also occur as the result of releases from sources that are not related to the mine in Libby (e.g., commercial products or natural sources). Also, some other minerals (e.g., pyroxenes) are sometimes difficult to distinguish from actinolite and tremolite (Bern et al. 2002). Because mineralogical data may or may not inform our understanding of the toxicity of LA, delineating amongst these mineral types is desirable at this stage of data collection. Therefore, the primary focus of this temporary modification is to collect more detailed data on the frequency of occurrence of sodium and potassium-containing particles both for samples from Libby and for samples from other locations.

#### Potential Implications of this Modification:

This temporary modification does not change any current procedures other than to require more detailed recording of data on particles observed under TEM. These additional requirements are not associated with a significant increase in time or cost of analysis. Hence, there are no negative implications of the modification.

Laboratory Applicabili	ty (circ e one): All	Individual(s	)	······································	
Permanent	Date(s): 09/12/2007 ( Analytical Batch ID: ication Forms - Attach legible  (Complete Proposed	le copies of appro	Section) Effe	ctive Date:	
Permanent Modifi	ication Forms – Maintain leg	rible copies of app	proved form in a bin	der that can be acce	essed by analysts
Data Quality Indicator	(circle one) - Please re	eference definitio	ns on reverse side	e for direction on se	electing data quality indicators:
Not Applicable	Reject	Low Bias	Estimate	High Bias	No Bias
Proposed Modification when applicable):	i to Method (attach add	litional sheets	if necessary; sta	ate section and	page numbers of Method
See Attachment 1					
Note: This modification	n (LB-000066c) supers	sedes LB-000	066b.		
Technical Review:	Laboratory Manager or d	esignate)	)	· · · · · · · · · · · · · · · · · · ·	Date:
Project Review and Ap	pproval: (Vplpe: Proje	ct Technical Le	ad or designate)	<u></u>	Date: 9/12/07
Approved By: USEP	A Project Chemist or des	ignate)			Date:9[11]07
					A Committee of the Comm

#### REFERENCES

Bern A, Meeker G, Brownfield I. 2002. Guide to Analysis of Soil samples from Libby, Montana for Asbestos Content by Scanning Electron Microscope and Energy Dispersive Spectroscopy. U. S. Geological Survey Administrative Report. October 17, 2002.

Meeker GP, Bern AM, Brownfield IK, Lowers HA, Sutley SJ, Hoeffen TM, and Vance JS. 2003. The Composition and Morphology of Amphiboles from the Rainy Creek Complex, Near Libby Montana. American Mineralogist 88:1955-1969

USEPA. 2005. EDS Spectra Characteristic Study for Libby-Type Amphiboles. Report prepared by Syracuse Research Corporation. Denver CO, for USEPA, Region 8, Denver CO. March 15, 2005.

#### DATA QUALITY INDICATOR DEFINITIONS

**Reject** - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

**Estimate** - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

**No Bias** - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

Lab Modification Form Revision 9 (9-2-96)

#### ATTACHMENT 1

- 1. Continue to classify structures as LA, OA, or C in accord with current procedures.
- 2. For all NAM particles that were "close calls" (i.e., they required careful assessment to determine they were not LA or OA), record the NAM particle on the bench sheet. Be sure to place a zero in the "total" column to ensure the particle is not counted as an asbestos fiber. NAM particles such as vermiculite, biotite, hydrobiotite, gypsum, titanium and other minerals that are clearly not amphibole should not be recorded.
- 3. For all particles that are recorded (including NAMs), use the structure comment field to record one of the following comments:

Code	Meaning
NaK	Na and K are both clearly present
NaX	Only Na is clearly present
XK	Only K is clearly present
XX	Na and K are not clearly present

4. For all particles that are recorded, whenever possible, use the structure comment field to identify a probable mineral classification. Use the designation "WRTA" (winchite/richterite/tremolite/actinolite) to indicate a particle that is consistent in morphology and chemical composition with a particle that is likely to have originated from the vermiculite mine in Libby. This will include most NaK particles and may include some NaX and some XK particles. It is unlikely that this will include any XX particles. For all other particles, use the following codes:

AC - actinolite

TR - tremolite

AT - actinolite/tremolite (too close to call)

AM - amosite

AN - anthophyllite

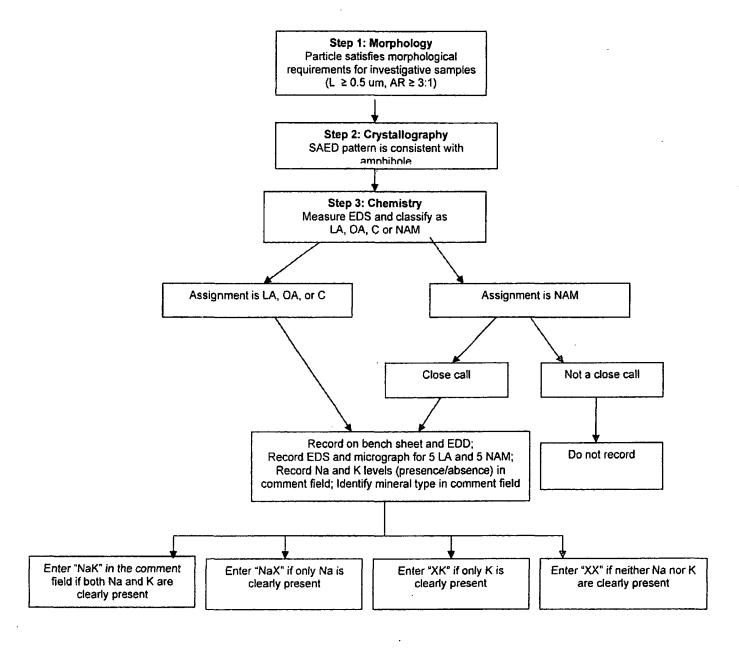
CR - crocidolite

PY - pyroxene

UN - Unknown

- 5. Increase the frequency that EDS spectra are recorded (saved). For each sample, record the EDS for each LA and each "close call" particle, up to a maximum of 5 LA and 5 "close call" particles per sample. To the extent practical, collect the EDS spectrum for a sufficient length of time that key peaks (e.g., sodium, potassium, aluminum), if present, can be clearly distinguished from background. Be sure that each EDS spectrum that is recorded can be linked to a specific particle in the EDD.
- 6. Increase the frequency that photomicrographic images of particle morphology are collected. For each particle for which an EDS spectrum is collected (up to 5 LA and 5 "close call" NAM, as discussed above), also record a photomicrograph of the same structures. Use the structure-specific comment field to record the photo identification number of each structure that is photographed. Convert all photographs to high quality electronic images (e.g., by scanning), and transmit the photos to CDM for evaluation.
- 7. Figure 1 provides a flow chart that summarizes the process implemented by this temporary modification.

## FIGURE 1 FLOW CHART SUMMARIZING THIS TEMPORARY MODIFICATION





## **Request for Modification**

# to Laboratory Activities LB-000085

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002	
EPA/600/R-93/116	
Other: All TEM and SEM Methods supporting Libby site investigative or Libby Action Plan (LAP)	
sample analysis	
Requester: Mary Goldade Title: Senior Environmental Scientist/Chemist	
Company: Environmental Protection Agency, Region 8 Date: April 2, 2008	
Description of Modification:	
Laboratories conducting transmission electron microscopy (TEM) or scanning electron microscopy (SEM) analysis	
support of either the Libby Site (all operable units, including Troy) or Libby Action Plan shall perform analysis of a	
reference standard to calibrate the energy dispersive x-ray spectrometry (EDS) analysis. The reference standard	<u>ı, а</u>
glass material referred as BIR-1G, was created by the USGS. It is recommended for use for Libby Amphibole analysis because it contains sodium (Na) and potassium (K) at known levels. Na and K are important elements u	ıcad
in Libby Amphibole identification by EDS. The BIR-1G standard was freezer-milled by EMSL to create particles for	
EM analysis. While generation of thin sections of the BIR-1G using a microtome was not feasible due to the expe	
analysis of the BIR-1G in particulate form is useful in standardizing the elemental measurements of the EDS and	
understanding the inherent variability in the EDS measurements.	
The BIR-1G shall be tested daily (on days that the TEM scope is used for analysis of Libby samples) and must m	<u>eet</u>
acceptance criteria prior to analysis of any field samples. Laboratories shall record the calibration information in	ا ــ : اـــ
accord with Attachment 1. As seen, not only does Attachment 1 provides the details for populating the electronic	aisi
deliverable (EDD) used in recording the calibration information, but Attachment 1 also describes the process for generating acceptance criteria for the BIR-1G standard for each individual instrument.	
generating acceptance officina for the Bit 10 standard for each manual instrument.	
Reason for Modification:	
The modification provides for a standardized process for performing and recording calibration standards for EDS	
during Libby Amphibole analysis.	
Potential Implications of this Modification: There are no negative implications to this modification. Positive impact	<u>:ts</u>
include a standardized process for: (1) daily calibration of a standard for the EDS used in Libby Amphibole	_
identification; (2) reporting results of BIR-1G measurements; and (3) generating acceptance criteria for the BIR-16 standard over time.	<u> </u>
Standard Over time.	
Laboratory Applicability (circle one): All Individual(s)	
Laboratory Applicability (circle one).	
This laboratory modification is (circle one): NEW APPENDS to SUPERCEDES	
Duration of Modification (circle one):	
Temporary Date(s):	
Analytical Batch ID:	
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages	
Dawnson (Complete Drangerd Medification Section) Effective Date: April 20, 2009	
Permanent (Complete Proposed Modification Section) Effective Date: April 30, 2008  Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.	
, comment industrial of the maintain regions copies of approved form in a small that our se account by analysis.	

Data Quality Indicator (circle one) - Please reference definitions on reverse side for direction on selecting data quality indicators: **Low Bias Estimate High Blas** No Bias **Not Applicable** Reject Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): N/A Technical Review: Date: (Laboratory Manager or designate) Project Review and Approval: \_ Date: (Volpe: Project Technical Lead or designate) Approved By: Date: (USEPA: Project Chemist or designate) DATA QUALITY INDICATOR DEFINITIONS Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable. Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low. Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

**High Bias** - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

**No Bias** - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

#### LB-000085

#### ATTACHMENT 1

#### Analyzing the BIR-1G Standard

- The BIR-1G standard shall be tested daily (on days that either the SEM or TEM microscope is used for analysis of Libby samples), prior to analyzing any field samples. Analyze for the compounds Na<sub>2</sub>O, MgO, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, K<sub>2</sub>O, CaO, TiO<sub>2</sub>, MnO, and FeO. It is suggested that the reference publication for BIR-1G be reviewed. It is available in Volume 2 of the Analytical Guidance Documents, Tab 35, provided by CDM.
- Set up TEM instrument and orient for typical Libby field samples.
- Record the TEM instrument details in the BIR-1G Electronic Data Deliverable (EDD) spreadsheet (see most recent version of Excel file "BIR-1G EDD.xls"). Note: Use one spreadsheet per TEM instrument.
- For each daily BIR-1G evaluation, select one particle and record the measured weight % for each compound as oxide weight % in the BIR-1G EDD. Note: When recording oxide weight %, enter results as a percentage not fractions (i.e., for 30%, enter 30 not 0.3).
- When selecting particles for analysis:
  - Choose particles in the middle of the grid opening and in the center of the grid.
  - o Particles should not be in close proximity to the grid bar or neighboring particles.
  - o Randomly select particles within different grid openings for each analysis.
- For selected particles, focus the beam on the thin edge, not the center of the particle.
- Continue analysis until a maximum peak height count of at least 1,000 is achieved for silicon (Si). This total Si count should be sufficient to achieve optimum instrument testing conditions. It is recognized that this total Si count may not be equivalent to typical analytical conditions for field samples.
- On a monthly basis, the EDD for each TEM instrument should be provided to EPA (or designated contractors).

#### Acceptance Criteria

- Acceptance criteria will be TEM instrument- and element-specific and will be derived from measured results.
  - $\circ$  Results that are within  $\pm 1$  standard deviation of the nominal will be ranked as acceptable.
  - Results that are outside  $\pm 1$  standard deviation but within  $\pm 2$  standard deviations of the nominal will be ranked as within the warning level.
  - Results that are outside ± 2 standard deviations of the nominal will be ranked as a failure.
- The potential bias of measured results will be assessed based on a frequency evaluation of results above and below the nominal.
- As needed, EPA will re-evaluate and revise the acceptance criteria to optimize program goals.

## **Corrective Action**

In the event that analysis results of the BIR-1G fall outside of the acceptance criteria, there should be a structured, progressive response. First, confirm that the detector/x-ray system has satisfied the acceptance criteria in the past. Next, confirm that the settings for the x-ray analysis software are correct (e.g. bias, scale). Finally, de-ice the LN2 dewar (unless it is a dry system) and carefully clean the window.

If these actions fail to rectify the problem, it will probably be necessary to send the detector/x-ray out to be serviced. The actions taken by the servicing company may include such things as baking the detector, renewing the vacuum in the dewar, checking the pre-amp or actual x-ray system for hardware defects, or replacing the crystal and/or FET (field effect transistor). In most instances the fault will not lie in the window unless the integrity of the window is compromised.

Upon the return and re-installation of the detector, re-run the BIR-1G standard to confirm that corrective action measures have resolved analysis issues.

# ATTACHMENT C GPS COORDINATES OF AMBIENT AIR SAMPLING LOCATONS

# ATTACHMENT C: GPS COORDINATES OF AMBIENT AIR SAMPLING LOCATONS

GPS Coordinate System: <u>UTM Zone 11N, NAD83 (datum), meters</u>

Station	Easting	Northing
A-4	617620	5366958
A-5	617797	5366301
A-6	618793	5365260
A-8	616466	5365187
A-9	617175	5365033
A-10	618317	5364708
A-11	618534	5366258
A-12	617157	5367325

## **TARGET SHEET**

# EPA REGION VIII SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 1075490

SITE NAME:	LIBBY ASBESTOS
DOCUMENT DATE:	
Due to one of the fo	DOCUMENT NOT SCANNED Illowing reasons:
☐ PHOTOGRAPHS	
☐ 3-DIMENSIONAL	
OVERSIZED	
☑ AUDIO/VISUAL	
☐ PERMANENTLY	BOUND DOCUMENTS
D POOR LEGIBILIT	·Y
□ OTHER	
□ NOT AVAILABLE	:
	MENTS NOT TO BE SCANNED Data Validation, Sampling Data, CBI, Chain of Custody)
DOCUMENT DESCR	IPTION:
1 CD - PART B: A ATTACHMENTS	MBIENT AIR AND GROUNDWATER. S A-C